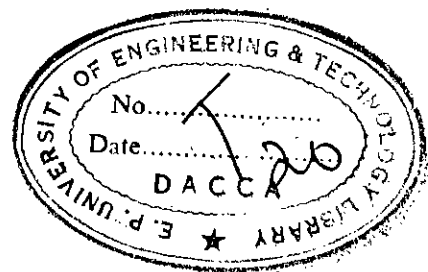


STUDIES OF FUNDAMENTAL PROPERTIES AND MECHANICAL BEHAVIOR OF
SELECTED SYNTHETIC AGGREGATES

A Dissertation

By

SUBODH KUMAR DAS



Submitted to the Graduate College of the
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Major Subject: Civil Engineering

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STUDIES OF FUNDAMENTAL PROPERTIES AND MECHANICAL BEHAVIOR OF
SELECTED SYNTHETIC AGGREGATES
(August 1966)

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ABSTRACT

Synthetic aggregates produced by firing clays and shales are being increasingly used in the construction of concrete and asphalt structures. The anomalous behavior of this synthetic product requires an extensive study of the material.

This research was a study of the changes in both mineralogical and physical properties of clays and shales during the process of firing. An attempt was made to explain a portion of the mechanical behavior of synthetic aggregates in relation to their fundamental properties.

The laboratory investigation was divided into two phases. During the first phase of this work, eight samples were selected to study the effects of temperature, rate and time of firing on bulk specific gravity and absorption characteristic of the fired materials. Two firing rates, designated as slow and rapid firing, were used for five samples. All eight samples were subjected to rapid firing for 8 and 15 minutes. A Globar furnace was used to fire the samples to temperatures ranging from 1200 to 2400°F. Mineralogical changes in all

samples fired under rapid firing for 15 minutes were studied using X-ray diffractions, differential thermal analyses, and petrographic examinations.

The second phase of this investigation was a study of the effects of firing temperature on microstructure, resistance to rapid freeze-thaw and abrasion, alkali-silicate reaction, compressive strength, and modulus of elasticity of the fired products. Two samples were studied in this phase of work. Several conclusions that were drawn from the test results follow:

1. New minerals that were formed by the transformation of clay minerals in the process of rapid firing were spinel, cristobalite, and mullite. The percentages of such crystalline minerals in the fired products were very low. Quartz, which was present in the unfired material, remained the predominant crystalline mineral in all synthetic products.

2. All bloaters indicated maximum bloating at about 2200°F and above this temperature they showed a reduction in bloating with the fusion of the material.

3. Minerals that dissociated at about 600°F, when present in sufficient quantity, caused bloating at temperatures around 2000°F where a viscous mass was formed under rapid firing.

4. Remolding of samples improved the bloating characteristics and formed a better cellular structure resulting in

lower absorption than the crushed samples.

5. As indicated by the ASTM Quick Chemical Test (ASTM Designation C289-64) of fired clays and shales, no deleterious properties of the fired material were caused by silicate glasses and crystalline minerals formed at high temperatures by the transformation of clay minerals.

6. For samples studied, resistances to freeze-thaw and abrasion, compressive strength, and modulus of elasticity showed appreciable variation with firing temperature. Resistance to freeze-thaw of fired products increased with an increase in firing temperatures from 1800 to 2200°F. Loss in abrasion was minimum at about 2000°F. Both compressive strength and modulus of elasticity of each sample indicated maximum values at about 1800°F with the formation of a vitrified or glassy bond within the material. Above this temperature, compressive strength and modulus of elasticity decreased rapidly with an increase in porosity of the material.

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For the uses of Globar furnace and X-ray equipment used in this work, the author expresses his thanks to Professor J.H. Caddess and Dr. K.J. Koenig of Mechanical Engineering and Geology Departments, respectively.

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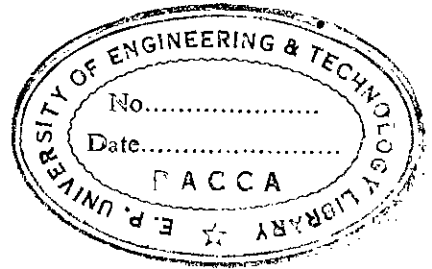
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CHAPTER I

INTRODUCTION

1.1. General Remarks

Experimental investigations on the production of synthetic aggregates over the last few decades have primarily been concentrated on the feasibility of producing lightweight aggregates through the transformation of particular clays, shales, and slates. But only limited works have been accomplished toward explaining their mechanical behaviors in relation to their fundamental properties. Investigations of the first type are no longer adequate to insure advancement in materials technology. Fundamental research, exploratory in nature and initiated by the desire of the researcher to discover unknown principles and facts, is necessary to continued technological advancement.

There is an utmost necessity for exploring the behavior of such material under different types of action, such as external load, thermal shock, impact, and many others. This knowledge will provide the basic methods to improve the usefulness of the material. With the knowledge of how and why materials behave as they do, it will be possible to make them tougher, stronger, and more durable.

1.2. The Problem in East Pakistan

Because of geological conditions, there are many regions in the world where natural deposits of sands and gravels used in the production of concrete and asphalt structures are either completely absent or only available in remote areas. As the need for aggregates has increased, this deficiency of natural aggregates is being substituted in many places by the use of synthetic aggregates produced from clays and shales.

East Pakistan, which is mainly an alluvial plain of the Brahmaputra and the Ganges Rivers, has a very limited source of natural aggregates suitable for highway, bridge, and building construction. Constructions in this country primarily utilize a crushed brick aggregate produced by firing clays to about 1200°F. Concrete produced from such aggregates, in most cases, possesses very poor strength and durability. For structures requiring high-strength concrete, it becomes necessary to transport natural aggregates from the north or southeastern part of the province. This is not only expensive but also extremely difficult because of the inadequacy of existing transportation systems.

Standing on the threshold of a rapidly developing world, it is absolutely necessary to solve some of the many problems yet confronting East Pakistan. The awareness of this necessity and need for advancement demand a very urgent utilization of her natural resources. It is, therefore, with a view to the

development of a feasible means of overcoming the problem of scarcity of natural aggregates in East Pakistan, that this research was initiated.

1.3. Objectives

The primary objectives of this research were:

1. To study the mineralogical changes in clays and shales in the process of rapid firing.
2. To study the effect of mineralogical composition of clays and shales on bulk specific gravity and absorption characteristics of the fired materials.
3. To determine the effect of firing temperature, rate, and time of firing on bulk specific gravity and absorption characteristics of fired clays and shales.
4. To study the effect of firing temperature on mechanical behavior of the fired materials.

CHAPTER II

HISTORICAL DEVELOPMENT OF KNOWLEDGE OF SYNTHETIC AGGREGATES

2.1. General Remarks

In 1918, Hayde (1)* produced the first commercial synthetic lightweight aggregate by expanding shale in a rotary kiln. Since then, it has been preferred in many constructions because of its certain advantages over natural gravel aggregates; particularly in its ability to reduce structural dead loads, to produce desirable insulating and acoustical properties, to increase fire resistance and -- in masonry units -- to give such properties as ease of handling and nailability. During the last few decades, investigations have been made into the conditions necessary for bloating clays and shales in the production of synthetic lightweight aggregates. Although many articles describing manufacturing processes and raw materials have been published in different journals, to the author's knowledge no significant work has yet been carried out to explain the basic mechanisms involved in the process of production and to correlate them with the engineering properties of the product.

*Numbers in parentheses refer to corresponding items in the list of references.

2.2. Conditions Necessary for Bloating Clays and Shales

Two conditions necessary for bloating of any clay or shale have been recognized by investigators for many years (2). These are:

1. The material must contain sufficient fluxing agents to produce a high-temperature glassy phase with a viscosity high enough to trap gas.
2. Some minerals that will dissociate to produce a gas at the temperature at which the glassy phase is formed, must be present.

Ceramists have long considered that bloating of clays is an adverse reaction in the ceramic process; only in the recent years has this bloating characteristic been utilized to produce a salable product.

Riley (2), by utilizing chemical analyses of a large number of bloating and non-bloating clays, was able to establish tentatively the compound limits on a composition diagram (Fig. 1). He reported that all clays, shales, and igneous rocks which had compositions that fell within the bloating area on the diagram bloated well. He also found that by changing the compositions of some non-bloating clays or shales with the addition of alumina and silica to fall within the bloating area, the materials could be bloated. While this was an important addition to the knowledge in this field, it should be pointed out that there were many clays which had chemical

compositons within the bloating area did not bloat satisfactorily (3).

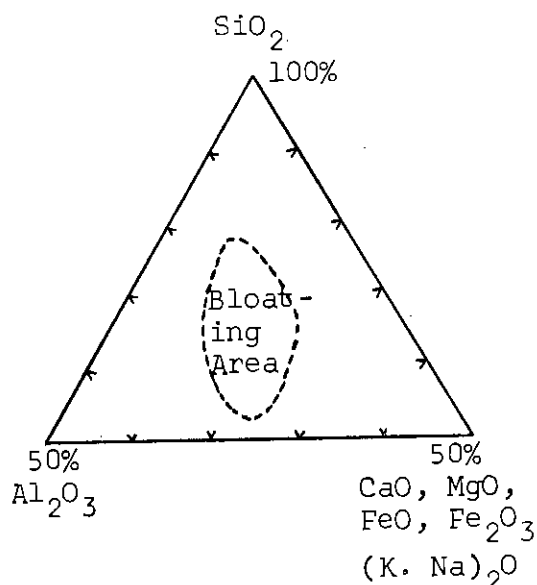


Fig. 1. Composition Diagram of Major Oxides Showing Area in Which Clays and Shales Fire to a Mass Viscous enough for Bloating (2).

2.3. Fluxing Agents in Clays and Shales

The most common fluxing agents in bloating clays and shales suggested by different investigators are CaO , MgO , K_2O , Na_2O , FeO , and Fe_2O_3 . Each of these agents has a definite temperature range within which it forms a viscous flux to trap gases liberated by the dissociation of gas-forming minerals within the material. For commercial bloating clays, this flux should be formed within the range of about 1800 to 2400°F and should persist through a range of at least about 200°F.

According to Riley (2), the limits of fluxing agents in a material necessary to produce a viscous mass within the temper-

ature range of 1800 to 2400°F are about 25 to 40%. Calcium is the common and active flux in most clays and shales. However, the bloating incidence of most calcareous clays is lower than any non-calcareous bloating clays. Thus, the presence of calcium in a clay or shale causes a better bloating under rapid firing than that under slow firing.

The high incidence of bloating among hydromica or illitic clays has been attributed to the presence of potassium. Grim (4) reported that a moderate amount of potassium in a clay increases the range in which a viscous mass is capable of entrapping liberated gases, and thereby increases bloating possibilities. The high incidence of bloating among the illite and montmorillonite groups of clays is primarily because of the presence of these fluxing elements as exchangeable cations and also as a part of the clay mineral structure. Absence of these exchangeable cations in kaolinite partly accounts for its low incidence of bloating.

Distribution of fluxing elements within the material has a great significance in the formation of a viscous mass proper for uniform bloating. Fluxing elements such as exchangeable cations or essential cations to the clay structure, as well as uniformly distributed, fine-grained accessory minerals, account for uniform viscous mass. Presence of fluxing elements as poorly distributed particles (such as calcareous concretions) cause intense fluxing action immediately adjacent to the particle resulting in a non-uniform viscous mass.

2.4. Gas-Forming Minerals in Clays and Shales

Many works have been reported by different researchers to explain the source of gases for expanding clays and shales in the process of heating. One of the first theories attempting to explain the mechanism of expansion of these materials at high temperatures was presented by Jackson (5) in 1903. He believed that this expansion was caused by the evolution of oxygen during the dissociation of ferric oxide contained in the raw material.

Orton and Staley (6) remarked that ferric oxide was not the only cause of bloating in all clays and shales. They suggested that if ferric oxide were the bloating agent in all cases then all clays and shales should bloat at the same temperature range. They showed that different clays and shales bloated at many different temperatures. They further noticed that sulfur dioxide produced by the dissociation of pyrite or marcasite present in the raw material caused bloating in many shales.

Wilson (7) reported that the evolution or expansion of sulfur dioxide or trioxide, carbon monoxide or dioxide, oxygen, entrapped air, and steam could cause bloating of clays and shales.

Conley, Wilson, and Kliniefelter (8) studied the bloating characteristics of a large number of clays, shales, and slates. They reported that the constituents responsible for the bloat-

ing of any material were minerals generally associated with clay minerals as impurities. These include carbonaceous materials, various iron compounds, limestone, dolomite, and gypsum which are potential sources of gases for bloating clays and shales. The presence of these materials is more frequent in shales than in clays. This is primarily because of the great compactness of shales which retards the leaching action of rain water, an agent for removing soluble gas producing compounds from clays.

They found that the temperature of liberation and retention of gases varied from about 900°F, accompanying the dehydroxylation of clay mineral, to 2400°F where some of the sulfates dissociated. They also discussed the dissociation temperature of different gas-forming minerals. Depending on the nature of the carbonates, the evolution of carbon dioxide begins at about 700°F and may continue up to about 1800°F. The dissociation of calcium carbonate takes place in the temperature interval 1100 to 1850°F. Magnesium carbonate alone or present in dolomite dissociates independently of calcium carbonate at temperatures from 1100 to 1400°F. Ferrous carbonate decomposes easily at about 700°F.

Sulfur, generally present in the form of pyrite, marcasite and calcium sulfate (CaSO_4), can evolve as sulfur dioxide in the interval 930 to 2450°F depending on the oxygen content of the surrounding gases, the temperature and the catalytic action

of the clay. The compound FeS_2 can change to either ferric or ferrous sulfate at 1100 to 1470°F, which in turn decomposes to the oxide with the evolution of SO_2 or SO_3 . The decomposition of CaSO_4 , when heated in an oxidizing atmosphere does not take place appreciably until 2210°F and becomes maximum at 2420°F.

Ferric oxide under normal conditions starts to lose oxygen at about 2370°F. With a reduction in oxygen content in the heating atmosphere, the dissociation can start at a temperature as low as 1830°F. In an atmosphere of 50 per cent each of CO and CO_2 , FeO_2 can convert to FeO at temperatures as low as 1050°F.

Riley (9) suggested that simple dissociation of dolomite could not cause the formation of gas for bloating. However, some reactions between dolomite and the other minerals present in the raw materials could take place forming compounds which dissociate at a higher temperature than that of the dolomite itself.

2.5. Factors Affecting Degree of Bloating

Some works have been reported about the factors affecting the degree of bloating of different clays, shales, and slates. The principal factors investigated were (1) time of retention at a given temperature, (2) firing temperature, (3) specimen size, and (4) grain size.

It has been found by different investigators (10) that for most materials the initial bloating took place within five minutes. After initial bloating ceased, no appreciable increase or decrease in bloating was observed with a longer retention time at a given temperature. Most of these investigators had used retention times of 5, 10, 15, and 20 minutes.

For most bloating clays, shales, or slates, the bloating increases up to a certain firing temperature depending on the composition of the raw material itself. This temperature for commercial bloated materials has been reported by different investigators (10) to lie in the temperature interval of 2000 to 2400°F. Above a certain temperature, the bloating gradually decreases with an increase in temperature because of the fusion of the clay mineral and escape of the gases through the less viscous flux.

Conley, Wilson, and Kliniefelter (11) reported that the smaller the particle size, the greater the bloating for the same time of retention and firing temperature. This is because the distance for the heat to travel in the small particle is less and also the surface per unit weight is greater. They remarked that with longer heat treatment there might be a tendency for the smaller particles to begin slagging and then to decrease in volume.

Lawrence (10) studied the effect of grain size on the bloating characteristics of different clays. He prepared spheres of different diameters from a material of three dif-

ferent grain sizes. He found that specimens made from coarser grain sizes gave better bloating. He remarked that while the overall heat treatment was the same for all similarly sized spherical specimens, the larger grained ones, having a lower surface area per unit volume, received less heat treatment and less penetration by oxygen; and hence, more of the gas-forming agent was available for bloating when a viscous flux was finally obtained.

CHAPTER III

CLAY MINERALS AND THEIR HIGH TEMPERATURE TRANSFORMATIONS

3.1. Clay Minerals

Clay minerals, as defined by the mineralogists, are fine-grained, flake-shaped crystalline products of weathering, essentially hydrous aluminum silicate with some instances of substitution of magnesium and iron for aluminum; and possess plasticity, cohesion, and ability to adsorb different cations, generally sodium, magnesium, potassium or calcium, within a wide range of moisture contents.

Mineralogists have classified clays and related minerals primarily into four groups:

1. Kaolin group, 1:1 lattice type
2. Hydrated mica or illite group, 2:1 lattice type
3. Montmorillonite or expanding lattice group, 2:1 lattice type
4. Fibrous clay group

Clay mineral structures have been explained in considerable detail by different investigators. Two fundamental components are involved in the atomic lattices of most of the clay mineral structures. The first component consists of silica tetrahedrons in which each silica atom is surrounded by four oxygen atoms arranged in the form of a tetrahedron with a silicon atom at the center. These silica tetrahedral groups are arranged to form a hexagonal network. In most clay mineral

structures, the tetrahedrons are so arranged that the apex oxygen atoms point in the same direction and the bases of all tetrahedrons are in the same plane.

The second component is built of two sheets of closely-packed oxygen atoms and/or hydroxyls in which aluminum, iron, or magnesium atoms are embedded in a octahedral co-ordination, so that they are equidistant from six oxygen atoms and/or hydroxyls. With aluminum, only two-thirds of the possible positions are filled to balance the structure and with magnesium, all positions are filled to balance the structure.

The structural components in some fibrous clay minerals are different from those noted above. These clay minerals resemble the amphiboles in their structural characteristics. The basic structural unit is composed of silica tetrahedrons arranged in a double chain. This structure is similar to that of the silica tetrahedral layer in layer minerals except that it is continuous in one direction only.

The kaolin mineral is composed of one silica layer and one alumina layer. The atoms in this mineral are arranged in parallel sheets with the large anions, oxygen and hydroxyl, coordinated around the small cations Si^{4+} and Al^{3+} . The Si-ions are surrounded as closely as geometrically possible by tetrahedral groups of four oxygen ions and Al-ions by octahedral groups of six oxygen and hydroxyl ions.

The hydrated mica or illite clay mineral is composed of two layers of silica to one alumina layer. Isomorphous substitution of Al for Si in the silicate layers causes charges to develop on the silica layers which are satisfied either by potassium atoms or by the positive charge on the alumina sheet. When K-ions satisfy the charges, the mineral is called illite and with Mg-ions the mineral is called vermiculite.

The montmorillonite mineral consists of two silica tetrahedral sheets tied together through a central octahedral coordinated sheet containing aluminum and/or magnesium, iron, and occasionally other elements. The minerals of this group have a unique, so-called expandible lattice, which has a variable c-axis dimension depending on the thickness of layers of water molecules between silicate layers. A wide variety of substitutions in the octahedral and tetrahedral positions is possible within the structure. These leave it with a net negative charge that is satisfied externally by exchangeable cations.

It may be mentioned that the foregoing structure of montmorillonite is not accepted by all mineralogists. An alternative structure was suggested by Edelman and Favajee (12) in which half of the apex oxygens of the silica sheets are inverted.

The fibrous clay minerals have not been studied so extensively. There are two types of clay minerals in this group:

sepiolite (hydrous magnesium silicate) and palygorskite (hydrous magnesium aluminum silicate).

3.2. High Temperature Phase Changes in Clay Minerals

The phase changes that take place in clay minerals in the process of slow heating have been extensively studied by different mineralogists in recent years by differential thermal analysis (DTA) and X-ray diffraction.

DTA patterns of most clay minerals show an endothermic peak in the region of 900 to 1400°F corresponding to the removal of the hydroxyl group from the clay minerals and exothermic peaks at about 1830°F and above which correspond to the formation of new crystalline minerals.

Until recently, it was generally believed that the dehydroxylation reactions of clay minerals occur topoaxially, that is, there was a three-dimensional similarity between the crystal structures of the starting material and product. In that reaction, each crystal of the starting material was changed into something approaching a single crystal of the product. The hypotheses regarding the change in the positions of anions and cations, i.e., the reaction mechanism, were based on three assumptions. These were (1) the loss of water occurred more or less uniformly from all parts of the crystal, (2) the Si-O parts of the crystal were more stable than the metal oxygen parts, and (3) the silica-rich material often

presumed to be formed as a subsidiary product was pure SiO_2 .

Taylor (13) in 1963 remarked that none of these assumptions was securely based and suggested an alternative approach to explain the phenomenon. He explained the high-temperature transformations of clay minerals by considering them as assemblies of close packed, or nearly close packed, oxygen ions with cations in the interstices. From this point of view, it may be expected that the relatively small cations such as Si^{4+} , Al^{3+} , Mg^{2+} , Fe^{2+} , Fe^{3+} , and especially H^+ would move more easily than the oxide ions. It may also be expected that oxygen ions that readily form water are more likely to be those that can most easily escape from the structure; e.g., from near external surfaces or from grain boundaries, and not necessarily those present initially as OH-groups. Taylor concluded that on this hypothesis, a typical dehydroxylation reaction involves three types of processes:

"(i) Loss of oxide ions from exposed or 'donor' regions, and their combination with H^+ ions from all regions, to form water. The donor regions are changed into pores.

"(ii) Cation migrations. These may be of two kinds. Firstly, all cations other than H^+ ions migrate from the donor regions into the remaining or 'acceptor' regions, and H^+ ions migrate in the opposite direction. Secondly, within the acceptor regions themselves, further migration may cause differentiation into Si-rich or Si-poor parts to occur.

"(iii) Changes in the type of oxygen packing in the acceptor regions, to complete the formation of the crystalline products. These last changes may occur throughout the acceptor regions, or only in a certain part of them."

The temperature at which these changes take place is different for different clay minerals. Even for the same clay mineral, the peak temperatures for endothermic and exothermic reactions differ considerably depending on the degree of crystallinity of the mineral. A highly disordered structure dehydrates at a lower temperature, whereas a highly organized structure dehydrates at a higher temperature. These temperatures also depend on the rate and time of heating.

Wahl and Grim (14) studied the high temperature phase changes in different clay minerals. Typical differential thermal curves of montmorillonite, kaolinite, and illite are shown in Fig. 2. For well-crystallized kaolinite, the dehydroxylation is complete at about 1100°F. In case of poorly crystallized kaolinite, this temperature falls to about 1000°F. In most kaolinites, the dehydroxylation occurs at a temperature between 1000 and 1300°F.

Many earlier investigators (15) reported that following the loss of hydroxyls, the kaolinite minerals changed into an amorphous mixture of alumina and silica. But electron microscope studies of this dehydroxylated material showed that kaolinite particles lose their regularity in c-axis direction and retain their hexagonal outline far above this temperature. This dehydroxylated material is known as "meta-kaolin".

Grim (16) reports that in the temperature interval following the loss of hydroxyl water and that of the new phase

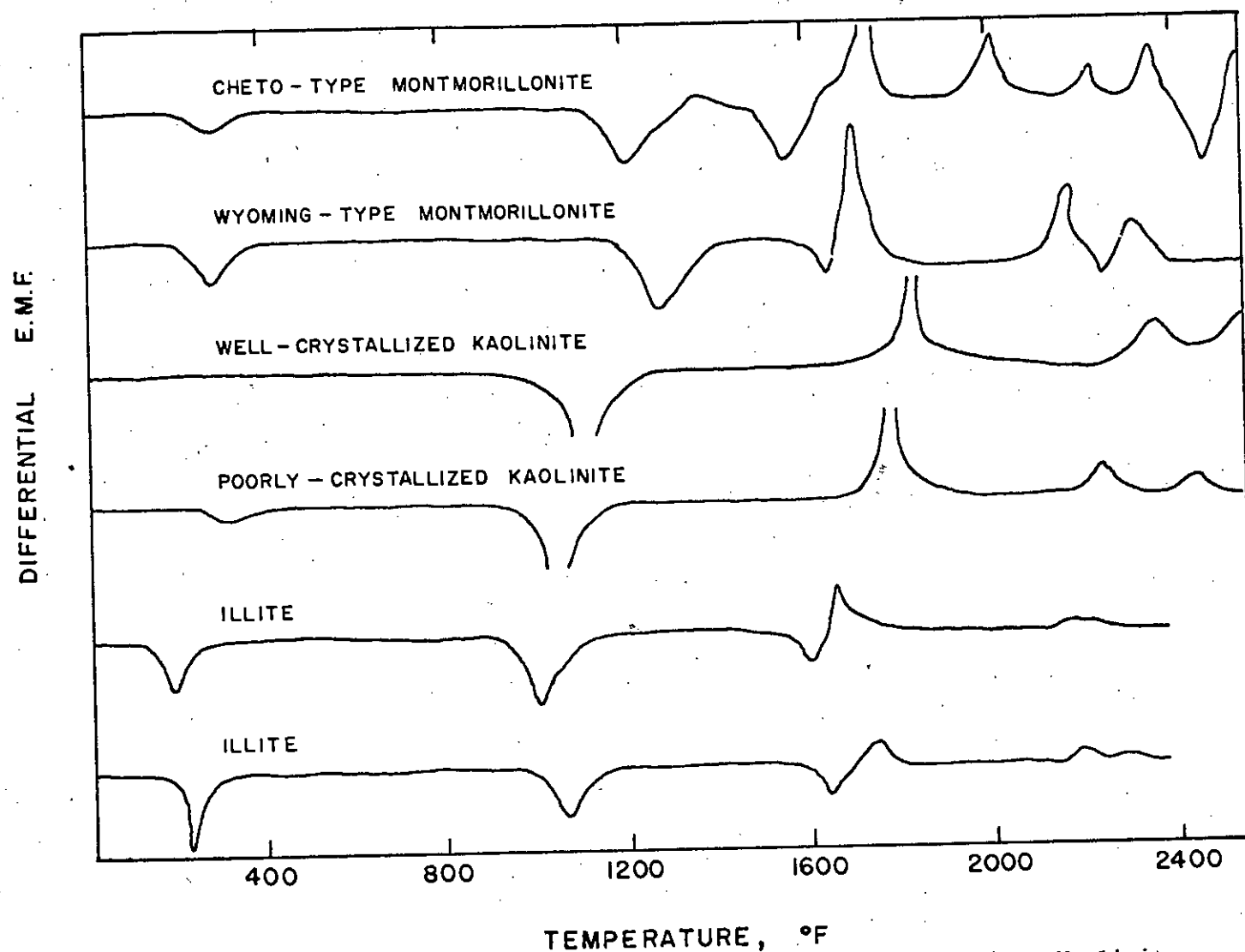


Fig. 2. Typical Differential Thermal Curves of Montmorillonite, Kaolinite and Illite (18).

development at about 1800°F, the silica and alumina are quite reactive. The alumina is readily soluble in acids and reacts readily with alkalies and alkaline earths.

The exothermic reaction at about 1800°F accompanies the formation of primary mullite. The second exothermic reaction at about 2300°F corresponds to the formation of secondary mullite. Wahl and Grim (17) found that the presence of primary mullite between 1800 to 2150°F is not in sufficient abundance to be detected on an X-ray diffractogram. The third exothermic reaction at about 2450°F has been attributed to the formation of beta-cristobalite.

The differential thermal curves of montmorillonite group of clays show the first endothermic reaction in the range of 210 to 570°F corresponding to the removal of interlayer water. At this temperature, the reaction is reversible, but becomes irreversible on further heating. The temperature at which such expanding clay minerals lose their ability to adsorb water depends primarily on the nature of adsorbed cation. Grim (18) reported that in montmorillonite this temperature is in the range of 190 to 435°F, 570 to 735°F and 735 to 915°F when lithium, calcium, and sodium, respectively, are the adsorbed cations.

The loss of hydroxyls from montmorillonites depends on the structure and composition of the mineral. For the dioctahedral form, the dehydroxylation occurs in the temperature

range of 840 to 1200°F, and for the trioctahedral form, this process is gradual and sometimes persists continuously to temperatures of the order of 1550°F without the loss of the original structure. In the case of dioctahedral montmorillonite, with a slight readjustment the general layer type of structure is able to survive even after the elimination of hydroxyl groups, and this anhydrite structure may persist to temperatures of the order of 1450 to 1650°F. DTA patterns on dioctahedral montmorillonite frequently show an endothermic peak in this temperature region which is probably related to the break-up of the anhydrite structure.

In the case of dioctahedral montmorillonite minerals, the dehydroxylated materials are not reactive between the temperatures of hydroxyl loss and first new phase formation, primarily because of its persistence of anhydrite structure. But for the trioctahedral type, the complete destruction of anhydrite structure results in a more reactive material.

The dehydroxylation of montmorillonite is followed by an exothermic reaction at about 1750°F with the formation of beta-cristobalite. In some montmorillonites, the formation of beta-quartz may be observed. Grim and Kulbicki (19) found that the formation of cristobalite could not be detected in an X-ray diffractogram until a temperature of approximately 2150°F was attained. The DTA pattern shows an exothermic reaction (sometimes more than one) at about 2200°F accompanying the

formation of mullite. Beyond 2200°F the pattern is too complex and variable to be interpreted with any degree of certainty. However, there is a suggestion of an exothermic peak between 2200 and 2400°F which may correspond to the development of cordierite, an endothermic peak at about 2300°F probably due to the break-up of cristobalite, and another endothermic peak little below 2550°F at the temperature of the beginning of the fusion of the sample.

The removal of the hydroxyl group from illite starts at about 850°F. In dioctahedral forms this process is comparatively rapid and takes place at temperatures in the interval of 850 to 1200°F. But in trioctahedral forms, it is very slow and may continue to about 1650°F, as in montmorillonite, where it shows a third endothermic reaction.

The loss of hydroxyl water in dioctahedral illite is not accompanied by a loss of structure. The anhydrite structure is destroyed at about 1550°F with the formation of spinel which increases in amount and particle size up through about 2200°F. Grim and Bradley (20) suggested that the octahedral layer of illite lattice carrying alumina, magnesia, and iron form spinel and that the alkali and silica from tetrahedral layers yield an amorphous material. At about 2200°F, spinel may turn into mullite which ultimately dissolves in glass at about 2550°F.

3.3. Effect of Impurities on High Temperature Phase Development

The high-temperature phase changes in clay minerals may be greatly affected by the presence of a very small amount of various impurities. Table I shows the effect of some impurities on the exothermic reaction of kaolinite.

TABLE I. The Effects of Impurity Additives on the Formation Temperature of Secondary Mullite and Beta-Cristobalite As Developed from Well-Crystallized Kaolinite (21).

Additive	Amount	Effect on Formation Temperature of Secondary Mullite	Effect on Formation Temperature of Beta-Cristobalite
FeCl_3	5%	Lowered 90°F	Lowered 180°F
MgCl_2	5%	Lowered 90°F	Lowered 180°F
CaCl_2	5%	Lowered 360-405°F	Lowered 336°F
LiCl_2	5%	Lowered 270°F	Prohibited phase
KCl	5%	Lowered 180°F	Prohibited phase
NaCl	5%	None	Prohibited phase
H_3PO_4	5%	None	Lowered 315-360°F

The effects of such impurities on montmorillonite and illite are not yet known. However, it may be remarked that the presence of impurities may affect the formation of any well-defined crystalline phase. Moreover, the presence of fluxes such as a large amount of iron, alkalies and alkaline earths may cause vitrification and fusion at temperatures as low as 1650°F (21).

CHAPTER IV

MECHANICAL PROPERTIES OF SYNTHETIC AGGREGATES

4.1. General Remarks

Under applied mechanical stresses, synthetic aggregates behave differently than metals. Metals, in general, have comparable tensile and compressive strengths, whereas synthetic aggregates are usually appreciably stronger in compression than in tension. This is mainly because of their differences in structure and the effect of structure on plastic deformation by shear.

4.2. Elastic Properties

With application of load, any material deforms. When the load is sufficiently small and the period of time is short enough, the deformation of an elastic material disappears with the removal of load. An excessive or long term load produces permanent atomic displacement or plastic deformation. Deformation which is recovered with the removal of the short term load is called elastic deformation.

With elastic deformation, the atoms keep their original neighbors and simply change their interatomic spacings. Unit stress required to produce unit elastic deformation is defined as the modulus of elasticity of a material. The resultant curve in Fig. 3 illustrates the variation of force (idealized) with

interatomic spacing in a material. Any change in the interatomic distance causes a significant change in the resultant force affecting the modulus of elasticity of the material.

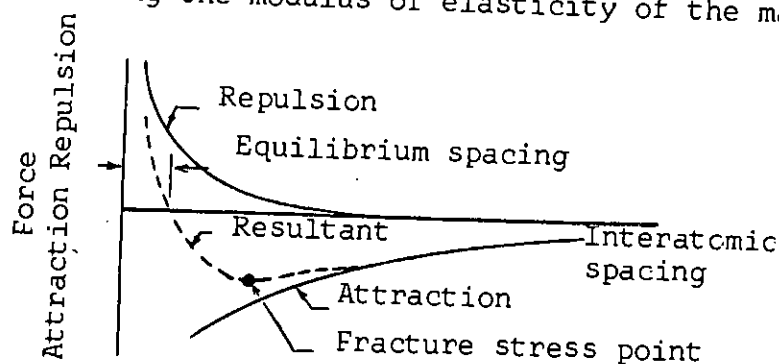


Fig. 3. Force Versus Interatomic Spacing (22).

It is known that the modulus of elasticity of a crystalline material may vary appreciably with crystallographic orientation. Since synthetic aggregates are a mixture of amorphous glass and polycrystalline phases with random grain orientations, the variation of modulus from grain to grain causes a non-linear stress-strain diagram. Those grains which are so oriented that they are strained in the direction of the lower modulus will have lower stresses than adjacent grains aligned in the direction of higher modulus. Since grains of different orientation will be deformed equally, some grains will undergo plastic deformation or fracture prior to other grains. As synthetic aggregates are polyphase materials, their elastic moduli depend on the elastic moduli of their different components and also on their arrangements (23).

4.3. Flow and Fracture of Synthetic Aggregates

The failure of a material under external load may be caused either by excessive deformation or by fracture. Since synthetic aggregates are essentially brittle, fracture is the more common mechanism of failure.

As synthetic aggregates are mostly a mixture of amorphous and crystalline minerals, they do not deform mainly by dislocation movements as crystalline materials do. Because of the absence of any definite crystal planes they do not exhibit any critical shear stress. However, both deformation and fracture can occur in such a material. Viscous flow can occur in the direction of maximum resolved shear stress. However, if the force is sufficient to break the highly stressed bonds in tension, fracture can be propagated at stress concentrations in directions normal to the resolved tensile stress (24).

The interatomic distances in a glassy material (Fig. 4) like synthetic aggregates are not uniform. Some spacing may be greater than the equilibrium spacing (Fig. 3) to a point near the fracture stress point. At this position, even a small external force can cause the failure of the bond. Once a bond has failed, neighboring atoms adjust their spacings, and subsequent bonds of higher spacing also fail. Hence, any persistent stress can cause continuous deformation resulting in a viscous flow.

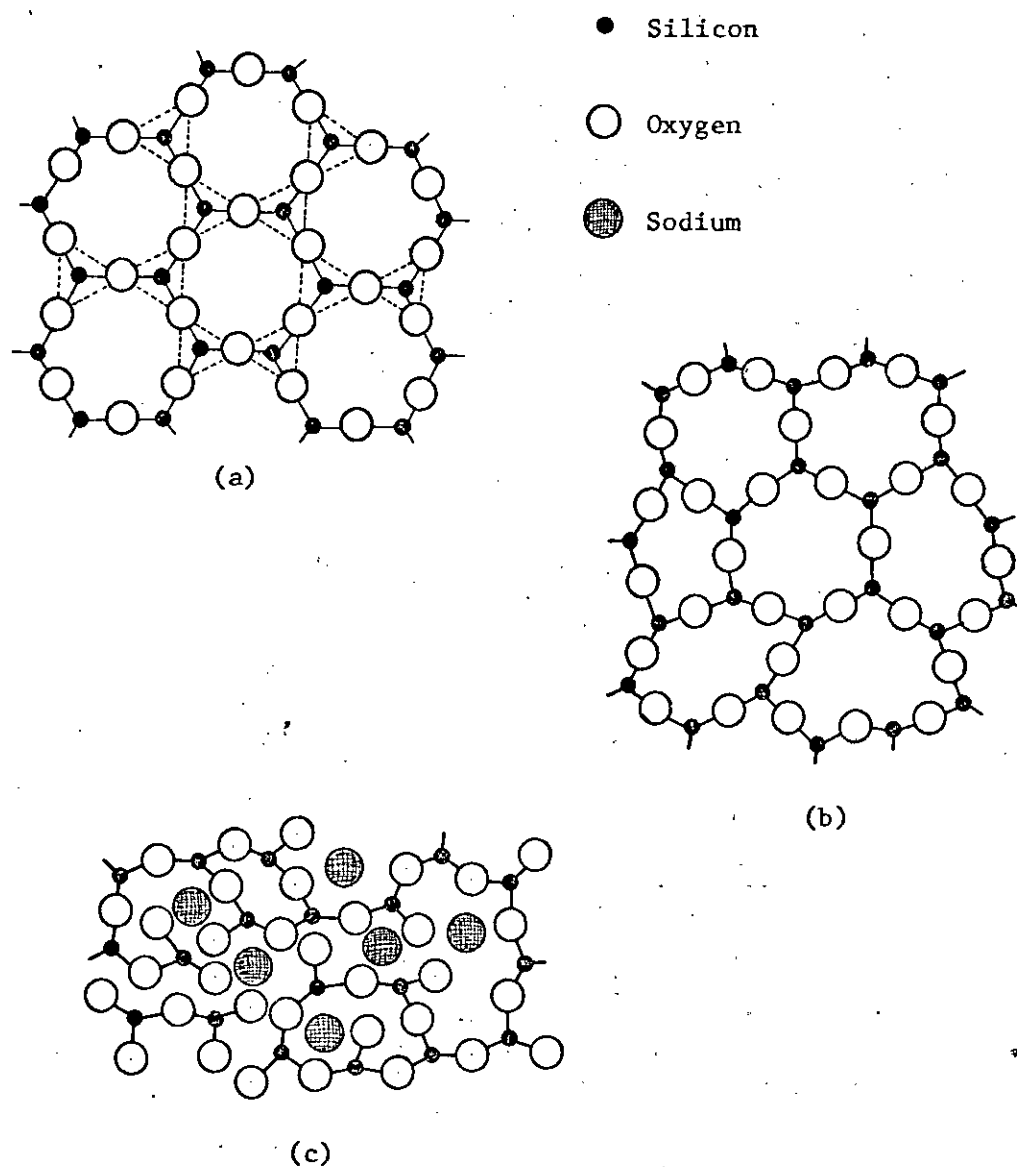


Fig. 4. Two-Dimensional Networks Analogous to Cristobalite and Silica Glass: (a) Crystalline Showing Network of Bonds (Full Lines) and Oxygen Triangles (Broken Lines); (b) Irregular; (c) Effect of Sodium Ions as in Soda-Glass (28).

The rate of deformation under an external load determines whether viscous flow or fracture of the material occurs. If the deformation rate is slow, after each bond is broken the neighboring atoms can adjust by viscous flow before additional bonds are broken. If the deformation rate is rapid, viscous flow cannot occur and after one bond is broken stresses are concentrated on the succeeding bonds resulting in fracture of the material.

4.4. Strength

A calculation based on interatomic forces would indicate that the strength of a material should be about 10 per cent of the modulus of elasticity (26). But, in general, this calculated value is about 10 to 100 times greater than the observed compressive strength of synthetic aggregates. Any rationalization of the strength of this material must account for this apparent disagreement. The microstructure of synthetic aggregates is such that there are many points where stresses become concentrated. These points are primarily pores, grain corners, or microcracks. Moreover, during the process of cooling of synthetic aggregates, stresses and strains are developed within the material because of their anisotropic properties in both thermal volume change and elastic moduli. Thus, there are many points of stress concentrations which finally result in initial cracks. Once a crack is initiated, it propagates rapidly through the material.

Coble and Kingery (27) studied the effect of porosity on creep (time-dependent deformation), strength, and elastic modulus of sintered alumina. They reported that the greatest effect of changing the amount of porosity was on creep properties, followed by strength and elastic moduli. Each of the properties tested showed that the maximum property values would be achieved at the maximum achievable density on structures in which the solid phase was continuous and contained isolated spherical pores.

CHAPTER V

TESTING PROCEDURES

5.1. General Remarks

The laboratory investigation was divided into two phases. In the first phase, a study was made on the effect of temperature, rate and time of firing on bulk specific gravity, and absorption characteristic of fired clays and shales. A study of the mineralogical changes in clays and shales in the process of rapid firing was also made in this phase.

The second phase was a study of the effect of firing temperature on selected properties of the synthetic aggregate products. These properties were bulk specific gravity, water absorption, durability against freezing and thawing, resistance to abrasion, potential reactivity, compressive strength, and modulus of elasticity.

5.2. Phase I

5.2.1. Materials. During the first phase of investigation, eight clay and shale samples were collected from different parts of Central Texas. Five of these raw materials are now being used in the production of commercial synthetic aggregates, and certain information concerning their engineering performances is available. Table II shows the physical properties of these samples.

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Table II. Physical Properties of Samples

Sample	Classification	Color	Texture	Reaction with Water	Remolded* Comp. Strength 24 hours at 230°F
ELD	Shale	Gray	Thin Lamination	Slake Slowly	830 psi
RGR	Shale	Gray	Thin Lamination	Slake Slowly	890 psi
DLS	Shale	Dark Gray	Thin Lamination	Slake Slowly	1050 psi
SFD	Clay	Dark Gray	No Lamination	Slake at Moderate speed	960 psi
CNV	Shale	Light Gray	Thin Lamination	Slake Rapidly	760 psi
EWD	Shale	Light Gray	Thick Lamination	Slake Rapidly	710 psi
SCT	Shale	Dark Gray	Thin Lamination	Slake Slowly	990 psi
WBN	Clay	Light Gray	No Lamination	Slake Rapidly	680 psi

*Each value average of three $\frac{1}{2}$ by $\frac{1}{2}$ by 1 in. briquette tests.

5.2.2. Preparation of Sample. About 30 lb. of each raw material was first dried and crushed to minus 16 mesh. From this sample, about 40 gm was separated and stored for X-ray analyses and differential thermal analyses. Water was added to the remainder of the sample and left soaking for 60 minutes to make it workable. Fifty-two 1 by 1 by 3 in. briquettes were made from the plastic mass of each sample. To avoid cracks from developing in the process of drying, it was necessary to air-dry the briquettes for three days. After three days of air drying, the samples were dried in an oven at 230°F for 24 hours.

5.2.3. Firing of Sample. The firing was carried out in a Globar furnace. Two firing rates were used for five of the samples designated as slow and rapid firing. Rapid firing only was used for the other three samples.

During slow firing, none of the samples was preheated, but was placed directly in the firing zone in the furnace. To avoid sticking of the melted material on the floor of the furnace, different specimen holders were tried and stainless steel trays were found suitable. Runs were made at 1200°F and then at 200°F intervals between 1800 and 2400°F. The average time to reach the run temperature from ambient was 38 minutes for 1200°F, 60 minutes for 1800°F, 75 minutes for 2000°F, 101 minutes for 2200°F, and 130 minutes for 2400°F. In the slow firing test, all samples were kept for 15 minutes at the desired

temperature of the run. Four briquettes were fired at each temperature.

During rapid firing, two different firing times, 8 and 15 minutes, were selected and samples were fired by subjecting them to temperatures of 1800, 2000, 2200, and 2400°F. Four briquettes were fired at each time-temperature combination. Despite predrying at 230°F for 24 hours, several early trials failed because the briquettes exploded when they were placed in the furnace at the firing temperature. It was therefore found necessary to keep the briquettes at about 900°F at least for 30 minutes before they were actually fired. This preheating was carried out in a different furnace.

In general, upon insertion of the material, there was an instantaneous drop in furnace temperature of about 60 to 80°F because of the heat capacity of the tray and briquettes. However, the temperature returned within about 60 to 90 seconds to that specified for the particular run.

Both slow and rapid firing of briquettes for the desired period were followed by rapid cooling to room temperature.

5.2.4. Specific Gravity and Absorption Test. Three briquettes of each run were broken into small pieces, ranging from 3/8 to 3/4 in. in size. Bulk specific gravity and 7-day absorption characteristics were determined by using the test method originally proposed by Saxer and then modified by Bryant (Appendix A).

5.2.5. X-ray Diffraction Analyses. Clay mineral composition of the minus two micron fraction of each sample was determined by using a General Electric XRD-5 X-ray diffractometer with nickel filter copper radiation generated at 35 Kv. and 15 ma. Specimens saturated with Mg, K, and Mg-ethylene glycol were prepared in thin films on glass slides. Per cents of different clay minerals were calculated by using the method outlined by Freas (28). Areas under (001) peaks which correspond to the c-axis distances of different clay minerals recorded from diffractograms made from oriented Mg-saturated slides were corrected by use of a factor of 1 for kaolinite, 3 for illite, 1 for montmorillonite, and 1/3 for chlorite. Per cent of each clay mineral was calculated as a per cent of total corrected areas.

5.2.6. Differential Thermal Analyses. The differential thermal analyses were made both on treated minus two micron fraction and also on untreated pulverized (minus 60 mesh) raw materials. The tests on untreated samples were carried out to study the cumulative effect of the impurities on the DTA pattern of treated samples and also to determine the dissociation temperatures of some of these impurities. The runs were made at about 21°F per minute for selected temperature intervals from room temperature to little above 1900°F. DTA patterns up to about 2400°F were desired, but laboratory facilities limited them to 1900°F.

5.3. Phase II

5.3.1. Preparation and Firing of Sample. During this phase, samples DLS and RGR were selected from the first phase of the study. About 100 lb. of each sample was dried and crushed to minus 3/4 in. sieve. Particles passing through 1/4 in. sieve were separated and stored for making briquettes to determine the compressive strength and modulus of elasticity of the fired material.

Firing was carried out in the same furnace used in Phase I. A stainless steel tray was used as the sample holder. Runs were made at 100°F intervals between 1800 and 2200°F using rapid firing with 15 minutes firing time. Ten pounds of each sample was fired at each temperature. To avoid explosion in the process of rapid firing, materials were kept at about 900°F for 30 minutes in a different furnace before they were actually fired. The immediate drop in temperature caused by the heat capacities of the tray and materials was about 100 to 110°F which was regained within about three minutes to that specified for the particular run.

5.3.2. Bulk Specific Gravity and Absorption Test. Bulk specific gravity (dry) and 28-day absorption characteristics were determined for the fired products of each run using the same method as in Phase I.

5.3.3. Abrasion Test. With the use of synthetic aggregate in road construction, the study of the resistance of this

material against abrasion or impact is of great interest. The Los Angeles abrasion test, which gives fairly good correlation with the actual wear of the aggregate used in concrete made with gravel aggregate, has been found to give erroneous results when used for synthetic aggregate, especially lightweight aggregate. A certain weight for sample depending on size, regardless of the weight-volume relationship of the material, is used in this test. Rushing (29) remarked that because of the lightness of synthetic aggregate the volume of the sample in the drum becomes high, which seriously affects the results due to cushioning effect.

Therefore, a method developed by the Texas Highway Department (Appendix B) was used in this test. Tex-404-A (30) test method was used to determine the unit weight of the material. The abrasion test was made for the fired product of each run. The percentage wear was determined by using both No. 4 and No. 12 sieves.

5.3.4. Freeze-Thaw Test. Because of the use of synthetic aggregates in exposed concrete and as coverstone, in recent years there has been a growing interest in studying the freeze-thaw resistance of this material. Meininger et al. (31) remarked that the determination of freeze-thaw resistance of aggregates may be useful in supplementing the freeze-thaw test of concrete specimens. They also stated that this type of test detects the non-durable particles in a heterogenous aggregate.

gate which provides a basis for separating out this fraction in a beneficiation process.

No standard test method has yet been developed to determine the freeze-thaw resistance of this material. A test method devised by Gallaway (32) was used in this work. The fired product of each run was washed and dried (24 hours at 230°F) and then divided into two fractions; group A, 3/4-1/2 in. size and group B, 1/2-1/4 in. size. The particles of each fraction were weighed and placed in an aluminum pan. The number of particles was approximately the number required to cover the pan one particle deep. Distilled water was added to the pan to the level about half particle deep, and this level was maintained throughout the test by adding distilled water from time to time. A chest-type freezer was used in this test. One freeze-thaw cycle consisted of about three hours of freezing at about $-3 \pm 2^\circ\text{F}$, followed by about 45 minutes thawing at $70 \pm 2^\circ\text{F}$.

After 50 freeze-thaw cycles, the samples were dried (24 hours at 230°F) and weighed. The per cent loss was calculated by using two different methods: (1) fraction passing through the sieve on which they were retained, $\frac{1}{2}$ in. sieve for group A and $\frac{1}{4}$ in. sieve for group B, and (2) fraction passing through No. 12 sieve.

5.3.5. Petrographic Study. The determination of relative amounts of crystalline and glassy material and the observation

of internal structure, size, shape, and mutual relationship of the mineral grains in fired products were made by studying thin sections with the aid of the petrographic microscope. Thin sections were prepared from both DLS and RGR samples fired at 1800, 2000, and 2200°F. The preparation of thin sections (about 30 microns thick) from these samples was extremely difficult because of their high degree of brittleness and the presence of impurities which caused poor compactness.

Three $\frac{1}{2}$ by $\frac{1}{2}$ by $\frac{1}{4}$ in. slices were obtained from each sample and impregnated at reduced pressure with Canada balsam dissolved in xylene. Xylene was then evaporated and the slices were allowed to cool. One face of each impregnated slice was ground smooth by using successively finer silicon carbide (carborundum) abrasives and was then mounted on a microscope slide with the smooth face in contact. With the use of successive finer abrasives on the other surface, the slice on the slide was then reduced to about 30 microns in thickness until minerals became noticeably transparent. A cover glass was then placed on each slide with cooked balsam dissolved in xylene as the cement. Excess balsam was washed away with xylene after three days. Microscopic study was made using both reflected and polarized light.

5.3.6. Potential Reactivity Test. In recent years, concrete distress attributable to alkali-aggregate (alkali-carbonate and alkali-silicate) reaction has been reported from

different parts of the world. But no rapid and conclusive test for determining the potential reactivity of an aggregate has yet been developed. Quick chemical tests have been recommended by ASTM designations C289-64 and C586-T (pending approval by ASTM) for silicate and carbonate aggregates, but they are not as reproducible as are ordinarily desirable. Mortar bar tests specified by ASTM designation C227-64 has a good correlation with field experience, but a considerable time is required before deciding whether an aggregate is reactive. In this work, the quick chemical test (ASTM designation C289-64) for potential reactivity of aggregates was used to determine the dissolved silica content. No test was made to determine the reactive carbonate content.

5.3.7. Test for Compressive Strength and Modulus of Elasticity. Ninety $\frac{1}{2}$ by $\frac{1}{2}$ by 1 in. briquettes were made from the plastic mass of each sample. Briquettes of larger sizes were tried, but they gave erratic results in the compression test because of the development of cracks in the process of both firing and cooling. Specimens were first air-dried for three days followed by oven drying at 230°F for 24 hours. After 30 minutes of preheating at about 900°F, runs were made of 1600°F and then at 50°F intervals between 1750 and 2000°F for a 15-minute firing time. Firing temperatures above 2000°F were tried, but excessive expansion occurred, distorting the samples, and was omitted. Ten briquettes of each sample was

fired at each temperature. To avoid cracks from developing in the process of cooling, it was necessary to cool the briquettes slowly in a steel box.

Three briquettes of each run were used to determine the porosity of the material. The bulk specific gravity was determined using the method stated in Phase I. The true specific gravity was obtained by measuring the specific gravity of pulverized material (minus No. 100 sieve) by a pycnometer technique.

Seven briquettes of each run were ground to make the ends flat and parallel. The dimensions of length and width of each specimen were measured within 0.001 inches. The compressive strength and modulus of elasticity were determined by using an Instron testing machine. The load-deformation characteristics of each specimen were measured by recording the load-time curve for the specimen and converting the recorded time axis to strain. The conversion was possible because of the extremely accurate rate of strain built into the machine. By equating the rate of strain of the machine to the time axis on the load-time curve, and correcting for the deformation of the machine under load, an accurate determination of strain was made. The modulus of elasticity of the material was computed by determining the slopes of the tangents to the stress-strain curves of each specimen at 25 per cent and 50 per cent of the ultimate strength.

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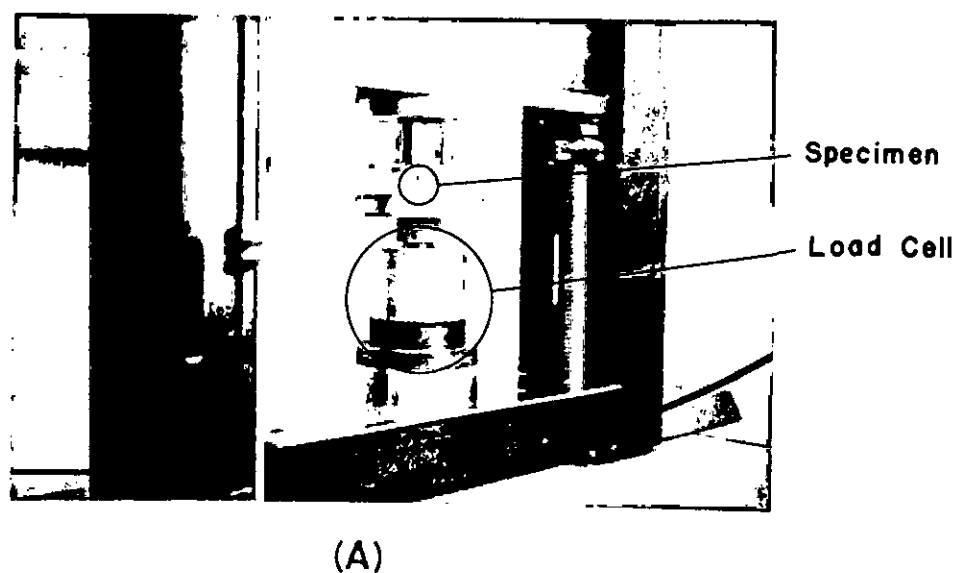
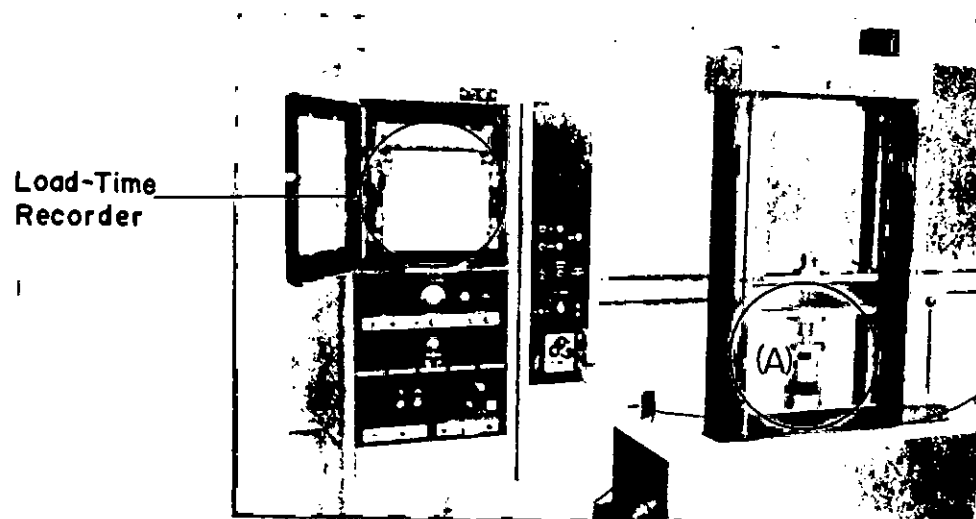


Fig. 5. Instron Testing Machine.

CHAPTER VI

RESULTS AND DISCUSSION

6.1. Changes in the Mineralogical Composition of Samples in the Process of Firing

To study the progressive changes in the mineral composition of each raw material in the process of rapid firing, both X-ray diffraction analyses and differential thermal analyses were used. Petrographic examinations were made to study the microstructure and mineral contents of fired products.

6.1.1. X-Ray Diffraction Analyses. Table III shows the clay mineral composition of eight samples. Changes in the crystalline minerals with firing temperature under rapid firing for 15 minutes are summarized in Table IV. In general, kaolinite minerals present in the raw material were destroyed at temperatures lower than 1200°F. The X-ray diffractograph of each sample showed the existence of illite and/or dehydrated montmorillonite at 1200°F. Figure 6 shows the typical X-ray diffractographs of sample SFD at different firing temperatures. The formation of new crystalline minerals by the dissociation of the clay mineral was first noticed in the diffraction patterns of all samples at 1800°F. Samples of RGR, ELD, and DLS containing illite with iron showed the formation of spinel at this temperature. Cristobalite was the first high temperature crystalline phase of montmorillonite in samples CNV,

Table III. Clay Mineral Composition of Samples.

Sample	Percentage of Different Clay Minerals					Bloating Characteristic & Initial Bloating Temperature
	Montmorillonite	Illite	Kaolinite	Chlorite	Chlorite & Illite Interstratified	
RGR	----	75	13	12	--	Very Good 2000°F
DLS	80	12	8	--	--	Excessive at Higher Tempera- ture, 1600°F
SPD	80	15	5	--	--	Good, 2000°F
ELD	----	55	12	11	22	Very Good 2000°F
CNV	100	--	--	--	--	Good, 2000°F
EWD	100	--	--	--	--	Poor, 2300°F
SCT	65	15	20	--	--	Excessive at Higher Tempera- ture, 1900°F
WBN	90	4	6	--	--	Poor, 2400°F

Table IV. High Temperature Crystalline Phases in Different Samples
After 15 Minutes Rapid Firing

Sample	Crystalline Minerals in Raw Sample	Crystalline Minerals at Firing Temperatures				
		1200°F	1800°F	2000°F	2200°F	2400°F
RGR	Chlorite, Illite, Kaolinite, Quartz, and Goethite	Illite, Quartz, and α -Fe ₂ O ₃	Quartz, Spinel, and α -Fe ₂ O ₃	Quartz, Spinel, and α -Fe ₂ O ₃	Quartz, Mullite, and α -Fe ₂ O ₃	Quartz and Mullite
DLS	Montmorillonite, Illite, Kaolinite, Quartz, Pyrite, Calcite and Felspar	Illite, and/or Dehydrated Montmorillonite, Quartz, Calcite and Felspar	Quartz, Spinel, Calcite and Felspar	Quartz, Spinel and Felspar	Quartz, and Mullite	Quartz and Mullite
SFD	Montmorillonite, Illite, Kaolinite, Quartz, Calcite, and Felspar	Illite and/or Dehydrated Montmorillonite, Quartz, Calcite, and Felspar	Quartz, Mullite, Calcite, and Felspar	Quartz and Mullite	Quartz, Mullite, and Cristobalite	Quartz, Mullite, and Cristobalite
ELD	Chlorite, Chlorite-Illite Interstratified, Kaolinite, Quartz, and Goethite	Illite, Quartz, and α -Fe ₂ O ₃	Quartz, Spinel, and α -Fe ₂ O ₃	Quartz, Spinel, and α -Fe ₂ O ₃	Quartz, Mullite, and α -Fe ₂ O ₃	Quartz and Mullite

Table IV. (Contd.) High Temperature Crystalline Phases in Different Samples
After 15 Minutes Rapid Firing

Sample	Crystalline Minerals in Raw Sample	Crystalline Minerals at Firing Temperatures				
		1200°F	1800°F	2000°F	2200°F	2400°F
EWD	Montmorillonite and Quartz	Dehydrated Montmorillonite and Quartz	Quartz and Cristobalite	Quartz and Cristobalite	Quartz and Mullite	Quartz and Mullite
SCT	Montmorillonite, Illite, Kaoli- nite, Quartz, Calcite, and Dolomite	Illite and/or Dehydrated Montmorillonite, Quartz, Calcite, and Dolomite	Quartz, Calcite, and Mullite	Quartz, Mullite, and Cristobalite	Quartz, Mullite, and Cristobalite	Quartz and Mullite
WBN	Montmorillonite, Illite, Kaoli- nite, and Quartz	Illite and/or Dehydrated Montmorillonite and Quartz	Quartz and Cristobalite	Quartz and Cristobalite	Quartz, Cristobalite, and Mullite	Quartz, and Mullite
CNV	Montmorillonite, Quartz, Dolomite, Calcite, and Felspar	Dehydrated Montmorillonite, Quartz, Dolomite Calcite, and Felspar	Quartz, Cristobalite, Calcite, and Felspar	Quartz, Cris- tobalite, Mullite, and Felspar	Quartz, Cristobalite, and Mullite	Quartz and Mullite

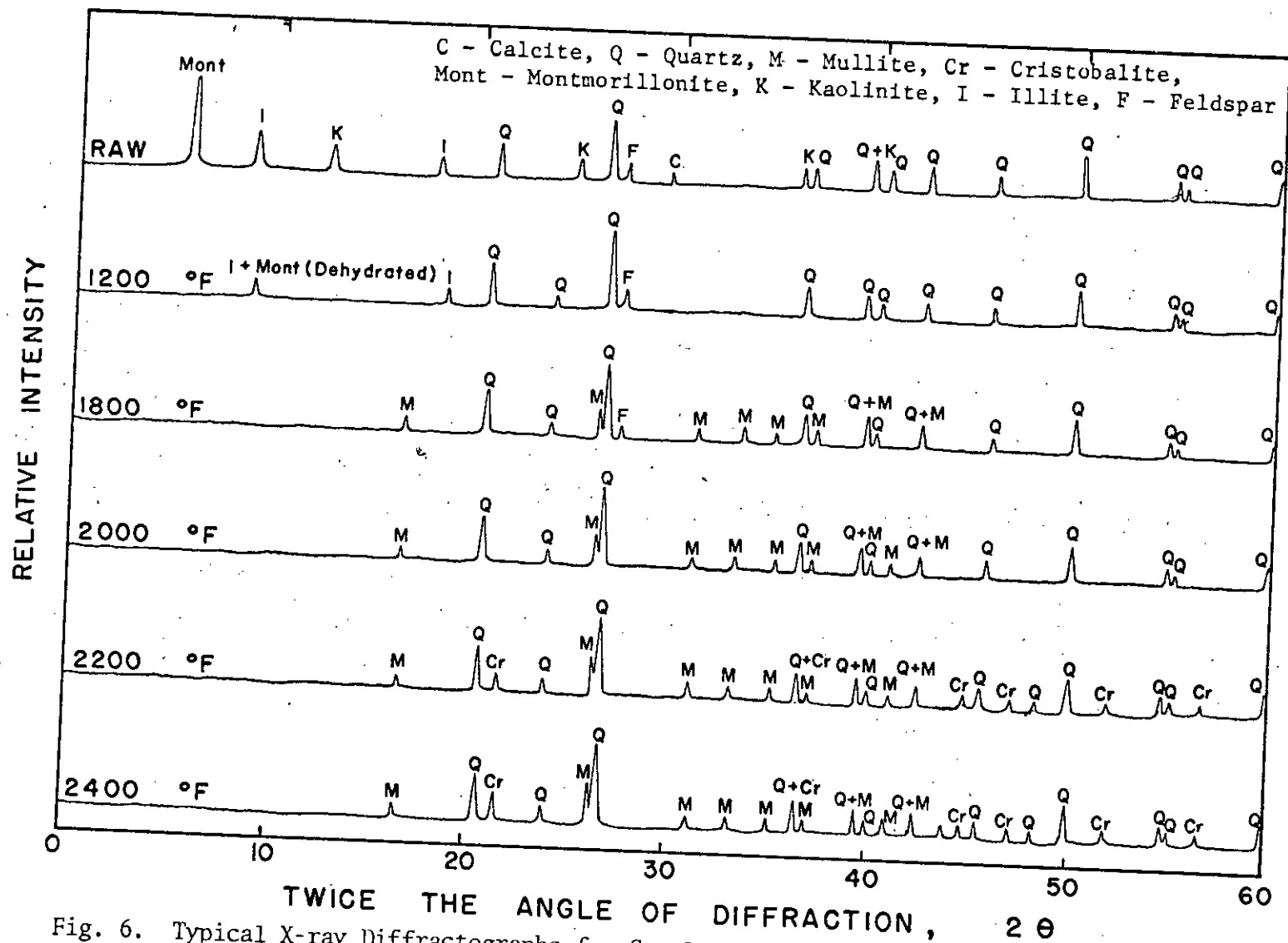


Fig. 6. Typical X-ray Diffractographs for Sample SFD Fired at Different Temperatures
 (Rapid Firing of Briquettes for 15 Minutes).

EWD, and WBN. Mullite was identified in the X-ray diffractographs of samples SFD and SCT at 1800°F. At 2200°F spinel was transformed into mullite in samples RGR, ELD, and DLS. Cristobalite disappeared at 2400°F from the X-ray diffraction patterns of all samples except SFD. Mullite was found in all samples fired at 2400°F. No significant change in quartz peaks was noticed in the process of firing except at 2200 and 2400°F where quartz peaks were weaker than at lower temperatures.

In addition to the foregoing clay minerals, some non-clay minerals which, in general, influenced the bloating characteristics were noticed in X-ray diffractographs of different samples (Table IV). These were goethite, pyrite, calcite, dolomite, and feldspar. Goethite present in samples RGR and ELD dehydrated to α -Fe₂O₃ below 1200°F and the α -Fe₂O₃ was finally decomposed at about 2200°F. Dolomite in samples CNV and SFD disappeared slightly below 1800°F. No calcite was observed at or above 2000°F. Feldspar was dissociated above 1800°F in sample SFD and above 2000°F in DLS and CNV.

6.1.2. Differential Thermal Analyses. Fig. 7 represents the DTA curves of seven samples. DTA patterns for samples EWD and WBN were similar. The first endothermic peak in each pattern at interval 212-284°F was accompanied by the removal of lattice water from clay minerals. The exothermic peaks ranging from 428 to 932°F in all samples except EWD were

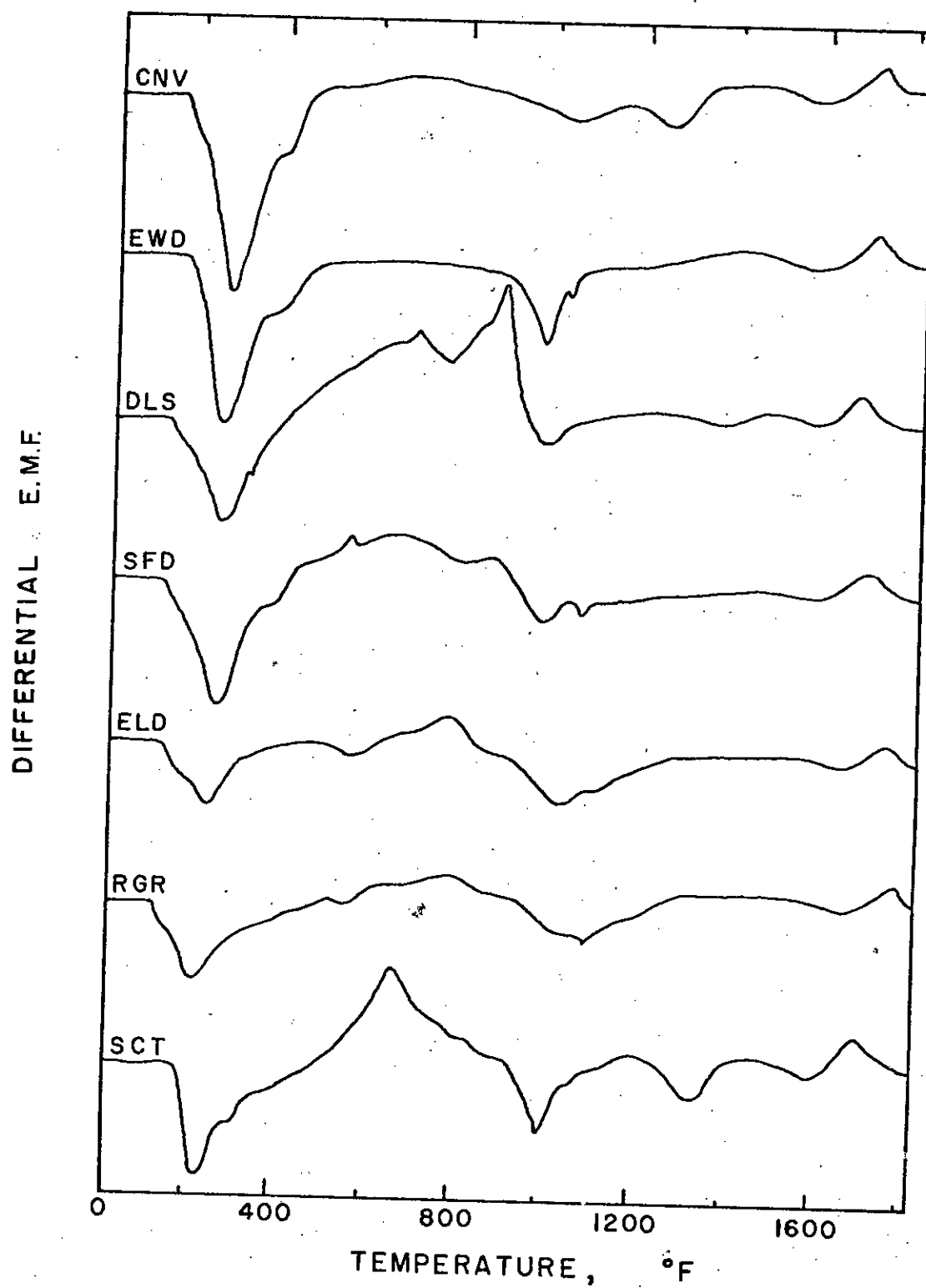


Fig. 7. Differential Thermal Curves.

formed by the burning of organic matter in these raw materials. Such organic matter was maximum in DLS, then SCT, SFD, RGR, ELD, and CNV. The dehydration of goethite in samples RGR and ELD took place at about 558°F as shown by the endothermic peaks in DTA curves of these samples. The exothermic peak at 882°F in sample DLS was accompanied by the decomposition of some of its pyrites or some sulfur associated with organic matter which was also detected by the odor of H_2S during slow firing of this sample. The dehydroxylation of different clay minerals was indicated by the endothermic peak at the interval 986 to 1058°F. The decomposition of dolomite in samples CNV and SCT was accompanied by endothermic peaks at 1256°F and 1346°F, respectively. The DTA pattern of each sample shows an endothermic peak between 1580 and 1670°F. This peak may be accompanied either by the prolonged dehydroxylation of trioctahedral montmorillonite and/or illite or by the decomposition of calcite. The first high temperature phase development in clay minerals took place at about 1688 - 1742°F as shown by exothermic peak in each pattern at this temperature. No DTA curves were obtained above 1840°F.

It should be stated that rate of heating of samples in this analysis was about 21.6°F per minute, whereas in actual firing of clays and shales in the furnace the heating rate in all cases was higher. Moreover, the sample used in the DTA was ground to minus 60 mesh which caused a uniform heating of

samples; but in the process of actual firing, materials were either remolded to 1 by 1 by 3 in. briquettes or crushed to 1/4 to 3/4 in. size, causing a non-uniform heating of each individual particle. These differences in rate of heating and particle size cause the existence of some minerals at temperatures above those of their dissociations indicated by DTA curves.

Differential thermal analyses on samples DLS fired at temperatures 1800 and 2000°F under rapid firing for 15 minutes indicated the presence of such undissociated organic matter and sulfur combined with organic matter. Dissociation temperatures of these materials are about 650 and 900°F, respectively.

A study of the clay mineral percentages in relation to bloating (Table IV) reveals that the problem of correlating mineral composition and bloating ability of a sample is complex. Figures 8 and 9 illustrate that some correlation exists between bloating characteristics of different samples having similar clay mineral compositions. Figure 8 shows that samples SFD and DLS, which have about the same clay mineral composition (Table IV), exhibit similar trends in changes of their bulk specific gravity and absorption with firing temperature. A similar correlation between samples RGR and ELD is illustrated in Fig. 9. From this study, it can also be remarked that no single mineral causes bloating of a given material. The minerals which, in general, contributed to bloating of a material

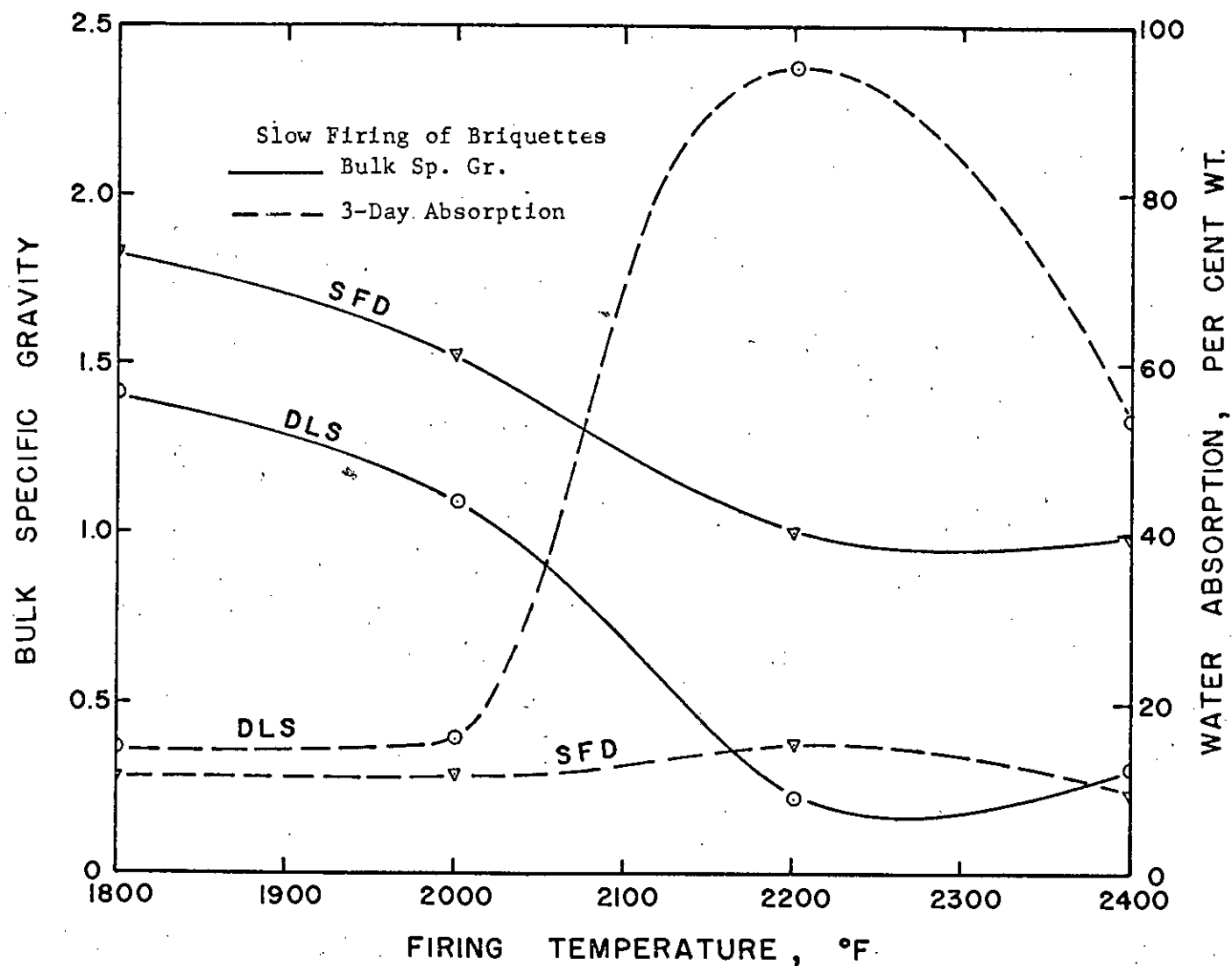


Fig. 8. Bulk Specific Gravity -- and Absorption -- Temperature Curves for Samples SFD and DLS.

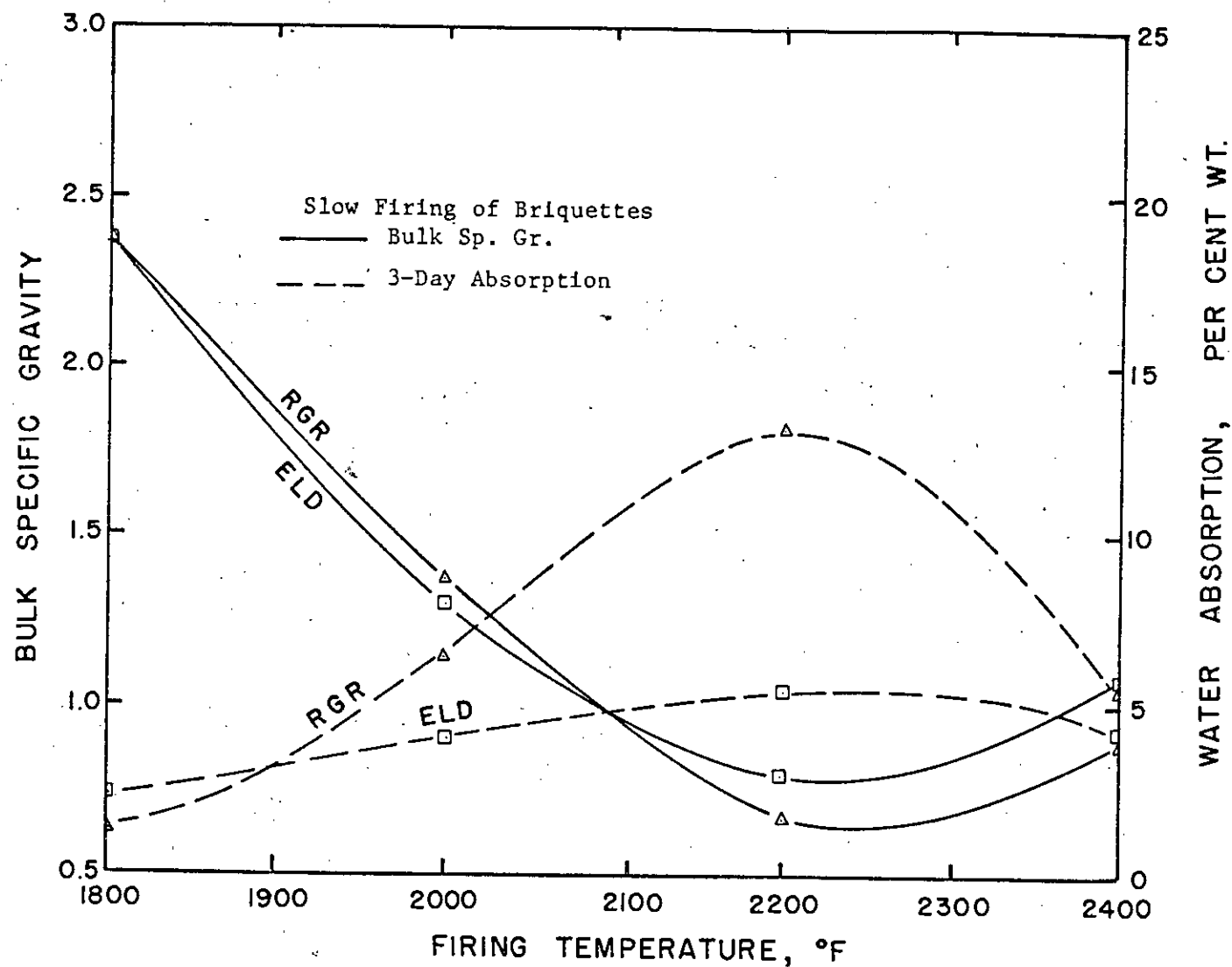


Fig. 9. Bulk Specific Gravity -- and Absorption -- Temperature Curves for Samples ELD and RGR.

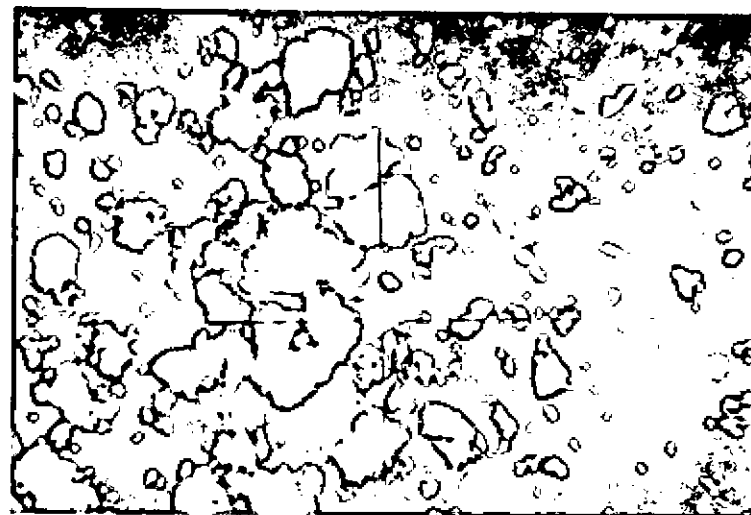
were kaolinite, illite, montmorillonite, chlorite, calcite, dolomite, pyrite, goethite, and organic matter.

6.1.3. Petrographic Studies. Figures 10 and 11 illustrate the microstructures of crushed samples of RGR and DLS fired at 1800, 2000, and 2200°F. Each sample was studied with both reflected and polarized light. Figure 10 shows the typical microstructure of thin sections viewed through a petrographic microscope with the Nicol prism partly crossed. In general, each picture can be divided into three parts: (1) the dark area which is the amorphous glass matrix, (2) the gray areas which are the pores, and (3) the white areas which are the crystalline particles.

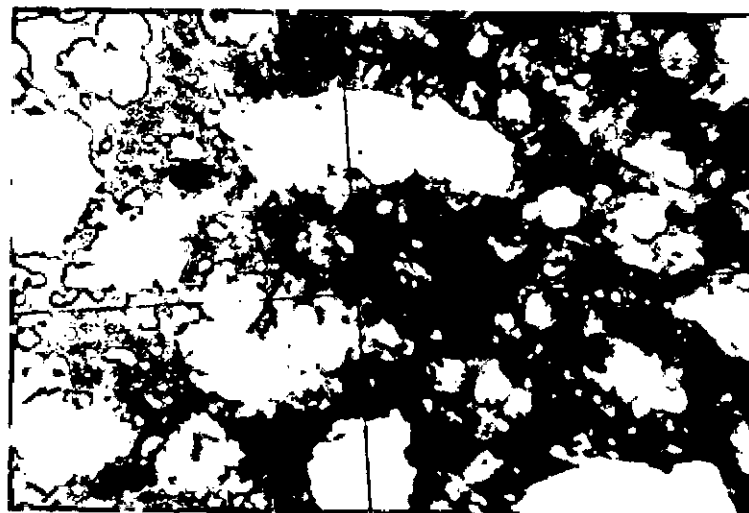
Sample DLS fired at 1800°F showed some silt size specks along with other characteristics indicate that the transformation process from raw shale to a fused glassy phase was incomplete, a process termed underburned. Such specks of red hematite were also observed in sample RGR fired at this temperature. Sample DLS appeared dark inside with a thin reddish-brown coating indicating very little or no oxidation inside the particle. Sample RGR was gray inside with a dense reddish-brown coating at the edge. At this temperature, both samples showed very low expansion. The pores were non-uniform and irregular. Silt size crystalline particles, mainly quartz, in each sample were angular with distinct edges. Table V illustrates the relative amount of pores, crystalline particles



1800 °F

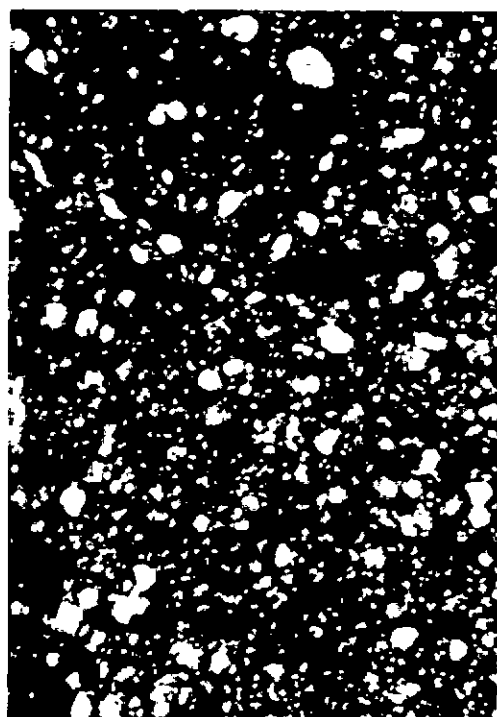


2000 °F

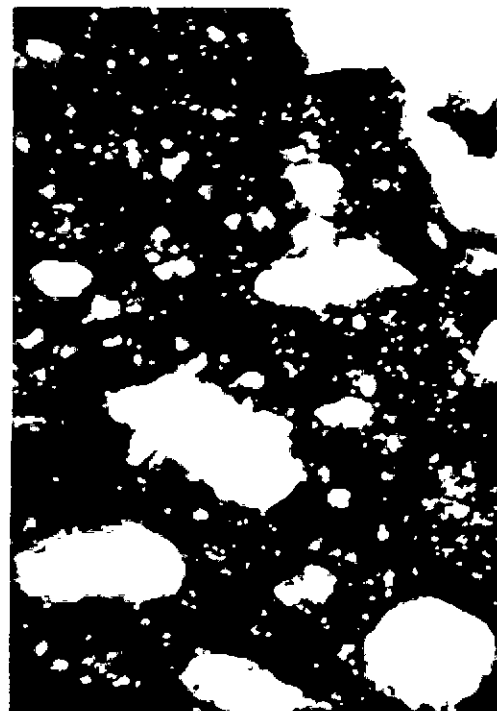


2200 °F

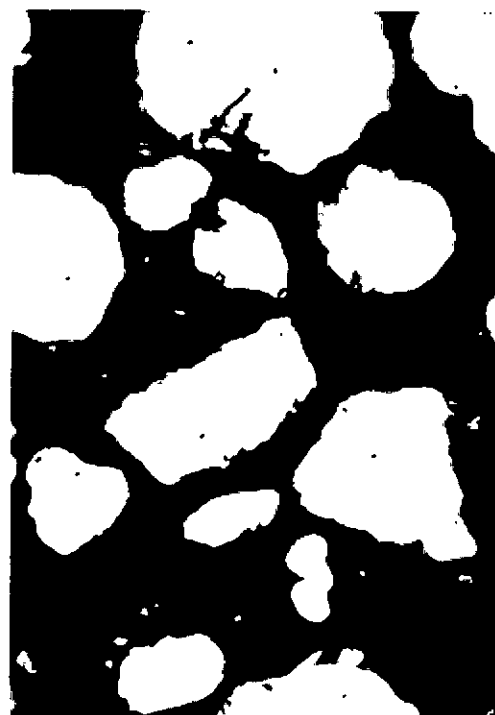
Fig. 10. Photomicrographs (80x) of Sample RGR. Nichol Prisms Partly Crossed. Black Areas Indicate Amorphous Glass Matrix, Gray Areas Indicate Pores and White Areas are Crystalline Minerals.



1800 °F



2000 °F



2200 °F

Fig. 11. Photomicrographs (100x) of Sample DLS. White Areas Indicate Pores and Black Areas Indicate Solid Matrix.

Table V. Size and Abundance of Pores and Crystalline Grains in Thin Sections of Samples RGR and DLS Fired at Different Temperature Under Rapid Firing.

Sample	Firing Temperature °F	Pores		Crystalline Grains	
		Size mm	Abundance Per Cent	Size mm	Abundance Per Cent
RGR	1,800	Very small to 0.2 x 0.7	6 - 8	Very small to 0.27 x 0.40	10 - 12
	2,000	Small to 0.5 x 0.7	15 - 18	Very small to 0.38 x 0.50	7 - 9
	2,200	0.12 x 0.15 to 0.6 x 1.3	30 - 40	Very small to 0.20 x 0.32	6 - 8
DLS	1,800	Very small to 0.4 - 0.7	11 - 13	Very small to 0.13 x 0.16	2 - 5
	2,000	Small to 0.6 x 0.8	22 - 26	Very small to 0.10 x 0.13	1 - 2
	2,200	0.28 x 0.82 to 1.5 x 2.7	55 - 65	Very small to 0.08 x 0.1	0 - 1

and amorphous glass matrix of each sample at different firing temperatures.

At 2000°F no FeS_2 was observed in any of the specimens. A few specks of red hematite were found in sample RGR, but both red hematite and unoxidized carbon particles were noticed in DLS specimen. The pores were more or less uniformly distributed and larger than those at 1800°F indicating considerable bloating at this temperature. The sharp edges of crystalline particles were partly fused and such particles were also diffused in the amorphous glass matrix.

At 2200°F sample RGR showed complete diffusion of most of its minerals. The pores were larger and still uniformly distributed throughout the glass matrix indicating good structural quality. Crystalline particles were mostly fused and reacted with the glass matrix. Sample DLS still appeared dark inside indicating incomplete oxidation of the material. The material revealed a large pore structure with interconnected cells.

From this study it is evident that in the process of rapid firing most samples fired at 1800°F contain a significant amount of undissociated material which dissociates even at lower temperatures under slow firing in presence of sufficient oxygen. Even under the same conditions of firing, the degree of burning varies from sample to sample depending on the nature of mineral contents of the sample. So, for different materials, different conditions may be necessary for a thorough burning

of each material. Moreover, rapid firing sometimes causes vitrification at the surface with underburning inside each particle. This underburning of clays and shales greatly affect its durability when used in concrete.

6.2. Firing Behavior of Samples

The effect of both slow and rapid firing on bulk specific gravity and absorption of different samples is summarized in Table V.

6.2.1. Bulk Specific Gravity. Figure 12 illustrates the change in bulk specific gravity of samples RGR, DLS, SFD, CNV, and ELD for a temperature range 1200 to 2400°F in the process of slow firing. The increase in bulk specific gravity exhibited in samples RGR and ELD indicates shrinkage up to a temperature about 1800°F. This increase in density of a material below its fusion temperature is a common mechanism. The process is known as sintering. It is a mechanism which reduces the surface area of loose particles below its fusion temperature, and the driving force arises from the excessive free energy of the surface of the loose particles over that of the dense material. Sintering results in the welding of loose particles resulting in a compact mass.

Figure 13 represents the change in true specific gravity of samples RGR and DLS. The increase in true specific gravity was greater for sample DLS than that for RGR. This was partly

Table VI. Physical Properties of Fired Briquettes

Table VI. Physical Properties of Fired Briquettes										
Sample	Firing Temperature °F	Slow Firing			Firing Time 8 Minutes			Rapid Firing		
		Bulk Sp. Gr.	24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent	Bulk Sp. Gr.	24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent	Bulk Sp. Gr.	Firing Time 15 Minutes 24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent
RGR	1,200	2.12	8.1	8.2	-	-	-	-	-	-
	1,800	2.36	1.3	1.3	2.39	2.3	-	-	-	-
	2,000	1.37	5.3	8.0	1.71	1.9	2.4	2.47	1.2	-
	2,200	0.66	10.9	15.3	1.12	3.5	2.3	1.66	1.8	1.3
	2,400	0.86	4.7	5.8	0.93	4.1	4.9	1.01	3.7	2.1
DLS	1,200	1.77	16.5	18.0	-	-	-	0.91	4.5	5.5
	1,800	1.41	11.5	17.7	-	-	-	-	-	6.5
	2,000	1.09	11.9	20.2	1.44	9.8	16.7	-	-	-
	2,200	0.22	75.5	112.2	1.19	9.2	16.1	1.43	9.3	-
	2,400	0.30	46.9	59.2	0.43	27.7	41.2	1.15	9.2	15.2
SFD	1,200	2.12	7.9	8.7	0.25	141.6	231.5	0.40	30.0	15.2
	1,800	1.83	9.1	11.8	-	-	-	0.21	156.6	45.0
	2,000	1.53	9.0	13.7	1.98	5.9	-	-	-	268.5
	2,200	1.01	11.2	19.1	1.74	6.8	8.7	1.91	6.4	-
	2,400	0.99	6.9	10.9	1.05	9.6	9.6	1.73	6.7	9.0
ELD	1,200	2.04	9.7	9.7	0.72	16.5	28.6	1.04	9.4	9.6
	1,800	2.37	2.3	2.4	-	-	-	0.65	17.4	14.6
	2,000	1.29	2.8	2.4	2.43	-	-	-	-	31.2
	2,200	0.79	4.3	5.3	1.76	1.7	1.9	2.48	1.3	-
	2,400	1.07	3.4	4.5	1.28	2.3	2.6	1.74	2.2	1.5

Table VI. (Contd.) Physical Properties of Fired Briquettes

Sample	Firing Temperature °F	Slow Firing			Rapid Firing					
		Bulk Sp. Gr.	Firing Time 8 Minutes		Bulk Sp. Gr.	Firing Time 15 Minutes		Bulk Sp. Gr.	Firing Time 15 Minutes	
			24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent		24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent		24-Hr. Absorp- tion, Per Cent	7-Day Absorp- tion, Per Cent
CNV	1,200	2.02	11.2	12.4	-	-	-	-	-	-
	1,800	1.69	4.4	5.7	1.65	4.1	5.3	1.64	3.9	5.0
	2,000	1.48	5.1	10.0	1.21	5.5	8.2	1.13	5.5	7.9
	2,200	0.96	6.9	11.6	0.56	5.9	9.6	0.51	5.7	9.3
	2,400	0.87	2.8	7.0	0.46	6.8	10.3	0.46	6.4	9.8
EWD	1,200	-	-	-	-	-	-	-	-	-
	1,800	-	-	-	2.22	2.5	2.9	2.23	2.4	3.0
	2,000	-	-	-	2.20	1.1	1.2	2.24	0.8	0.8
	2,200	-	-	-	1.59	1.7	2.1	1.56	1.2	1.9
	2,400	-	-	-	0.98	2.2	2.9	0.95	1.8	3.2
SCT	1,200	-	-	-	-	-	-	-	-	-
	1,800	-	-	-	1.69	8.7	10.9	1.63	9.1	11.5
	2,000	-	-	-	1.18	14.1	18.6	1.08	15.2	19.8
	2,200	-	-	-	0.29	43.6	66.9	0.28	47.5	71.0
	2,400	-	-	-	0.23	111.3	160.2	0.24	117.8	166.8
WBN	1,200	-	-	-	-	-	-	-	-	-
	1,800	-	-	-	2.05	3.1	4.9	2.06	22.8	4.4
	2,000	-	-	-	2.02	3.2	4.2	2.01	3.0	3.7
	2,200	-	-	-	1.73	3.6	4.7	1.64	3.2	4.9
	2,400	-	-	-	1.11	1.9	3.6	1.07	1.4	2.9

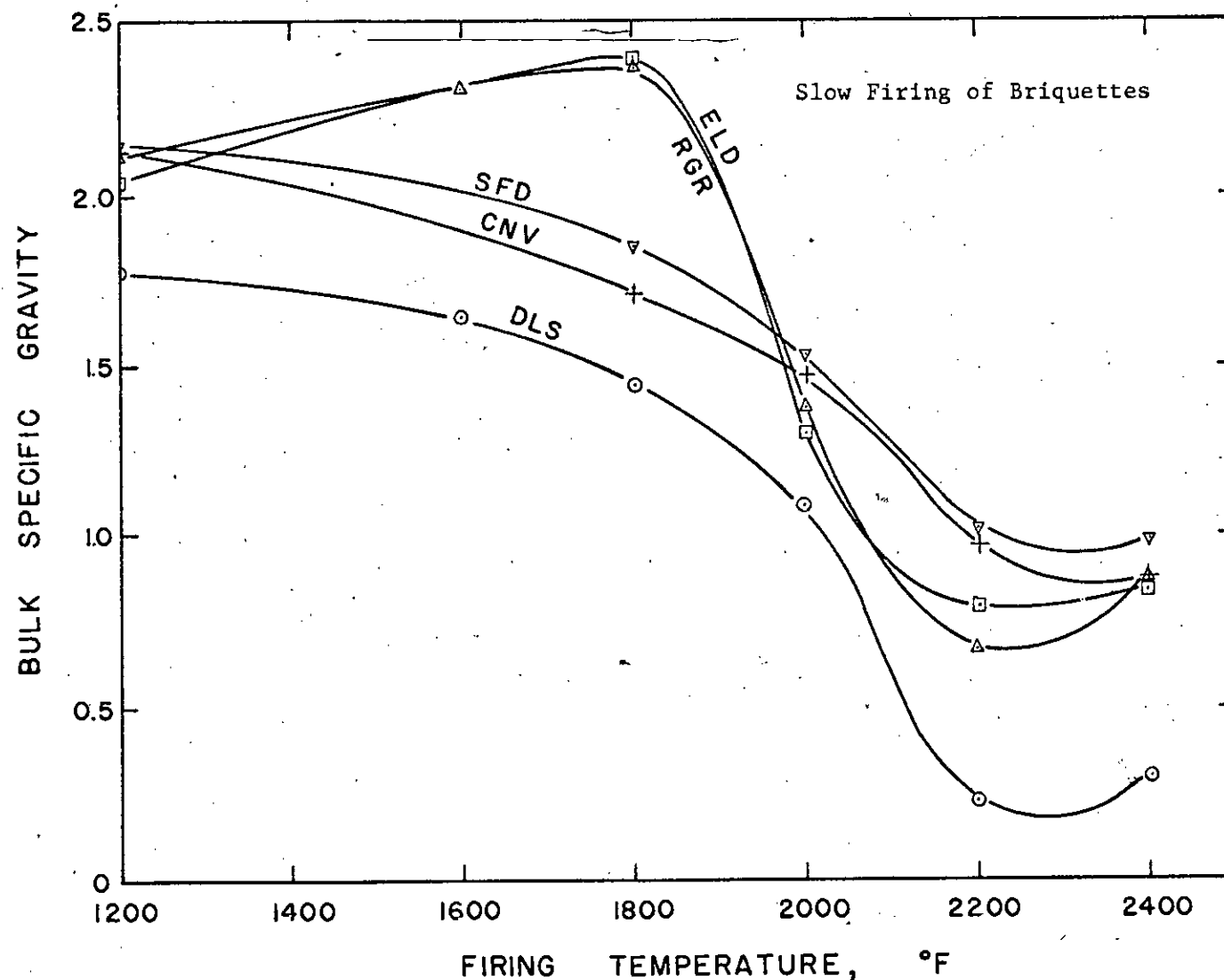


Fig. 12. Effect of Firing Temperature on Bulk Specific Gravity for Samples RGR, DLS, SFD, CNV, and ELD.

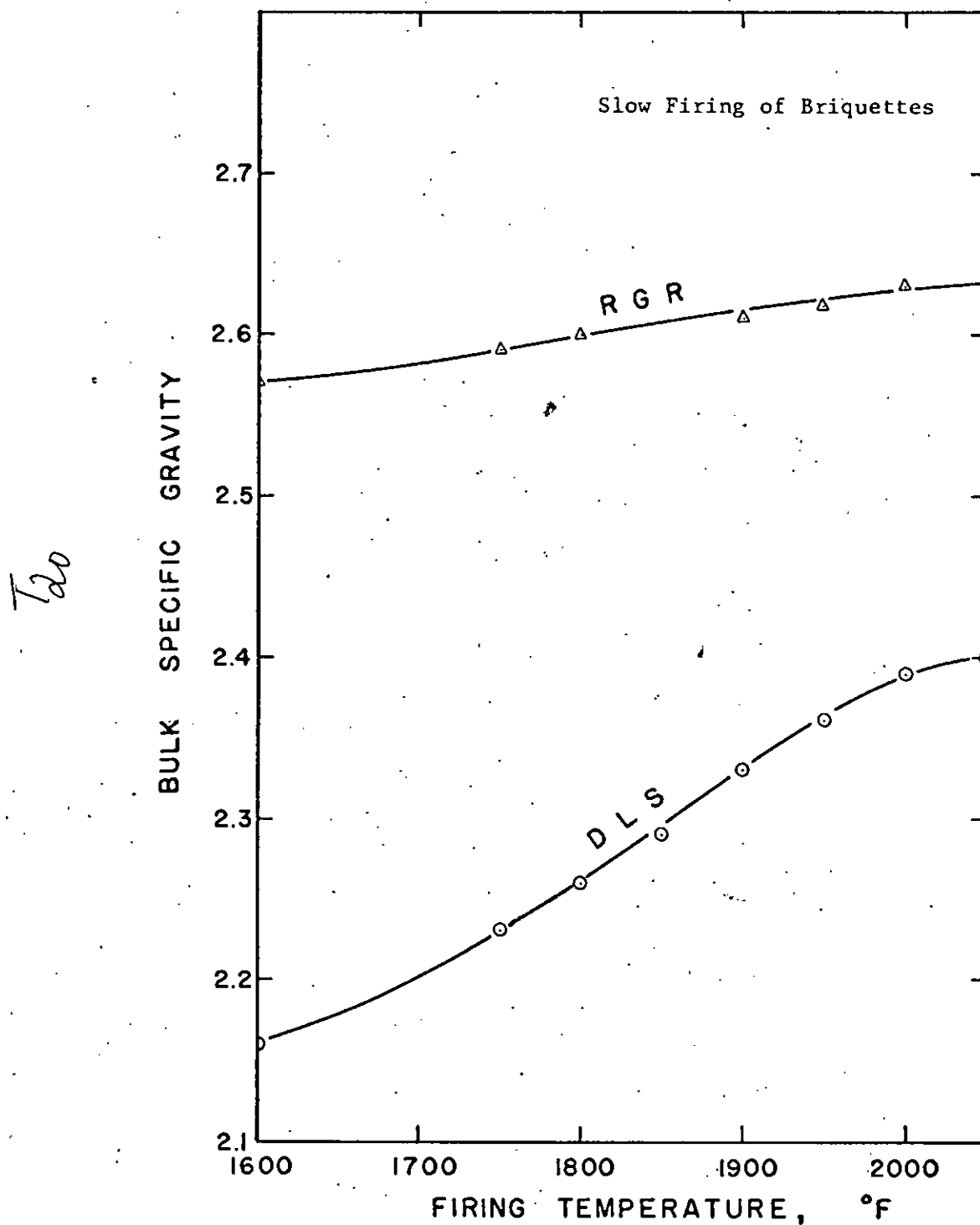


Fig. 13. Effect of Firing Temperature on True Specific Gravity for Samples RGR and DLS.

because of the presence of higher amount of organic matter in samples DLS. In each sample the increase in true specific gravity was decreased at temperature about 2000°F with the vitrification of some of its minerals. However, the formation of gases in samples DLS, SFD, and CNV at a relatively low temperature resulted in a decrease in bulk specific gravity. In general, all five of these samples showed maximum bloating at about 2200°F. The bloating was maximum in sample DLS, then RGR, ELD, CNV, and SFD. At temperatures above 2200°F, each sample began to fuse and the flux became less viscous, causing the mass to become permeable to gases. As a result the bulk specific gravity of the samples was noticed to increase with the escape of some of the entrapped gases above 2200°F.

Figures 14 and 15 illustrate the differences in bulk specific gravity of samples RGR, ELD, DLS, SFD, and CNV in the process of slow and rapid firing. In both cases, samples were retained at the desired temperature for 15 minutes. The changes in bulk specific gravity of samples RGR, ELD, DLS, SFD, SCT, and EWD under rapid firing for 15 minutes are depicted in Figs. 16 and 17. Samples ELD and RGR experienced lower bloating in the process of rapid firing, whereas samples DLS, SFD, and CNV showed higher bloating in this process. This increase in bloating was maximum in CNV, then SFD and DLS. It may be noticed in Figs. 14 and 15 that the maximum bloating in these three samples under rapid firing took place at

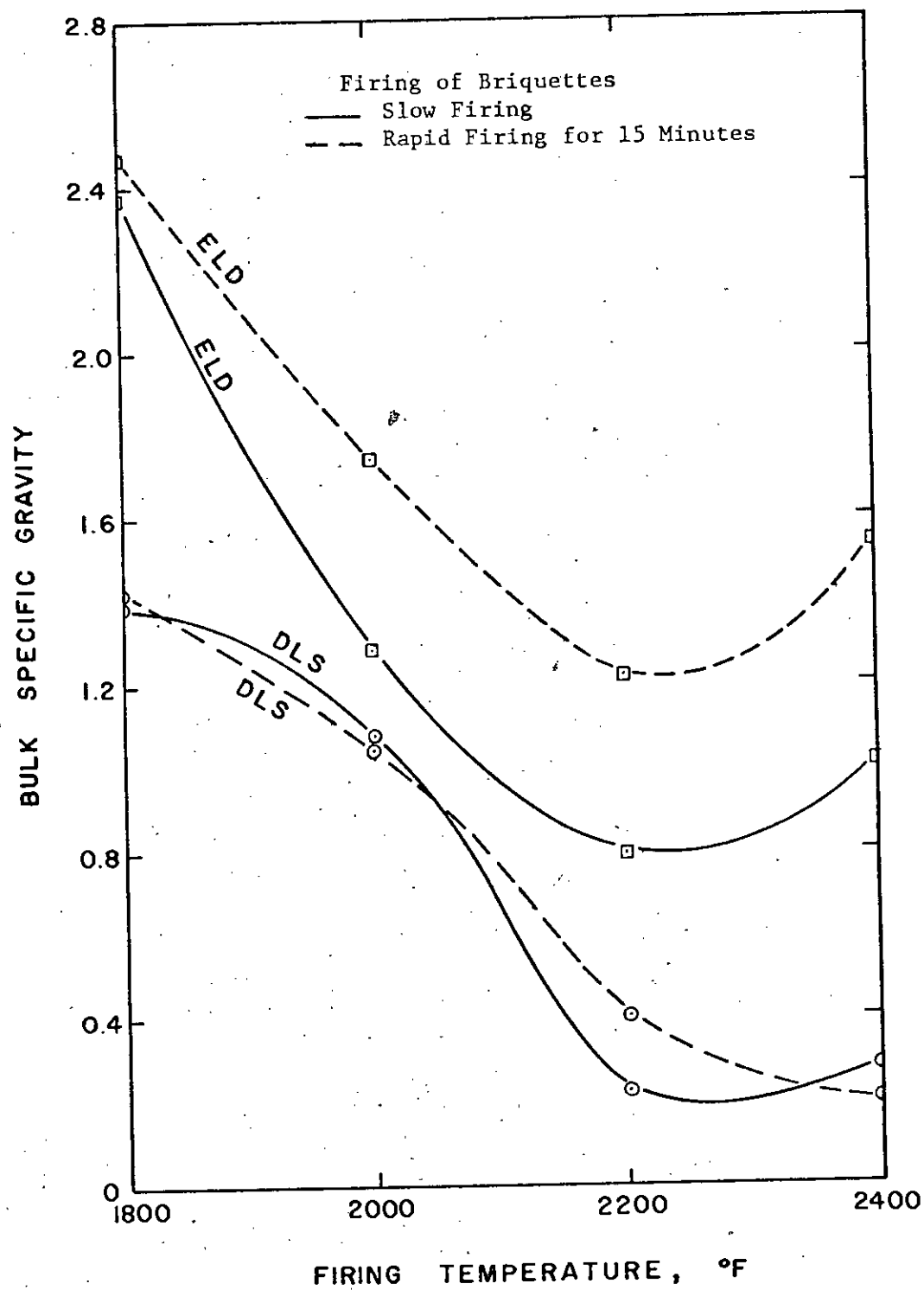


Fig. 14. Effect of Firing Rapidity on Bloating for Samples ELD and DLS.

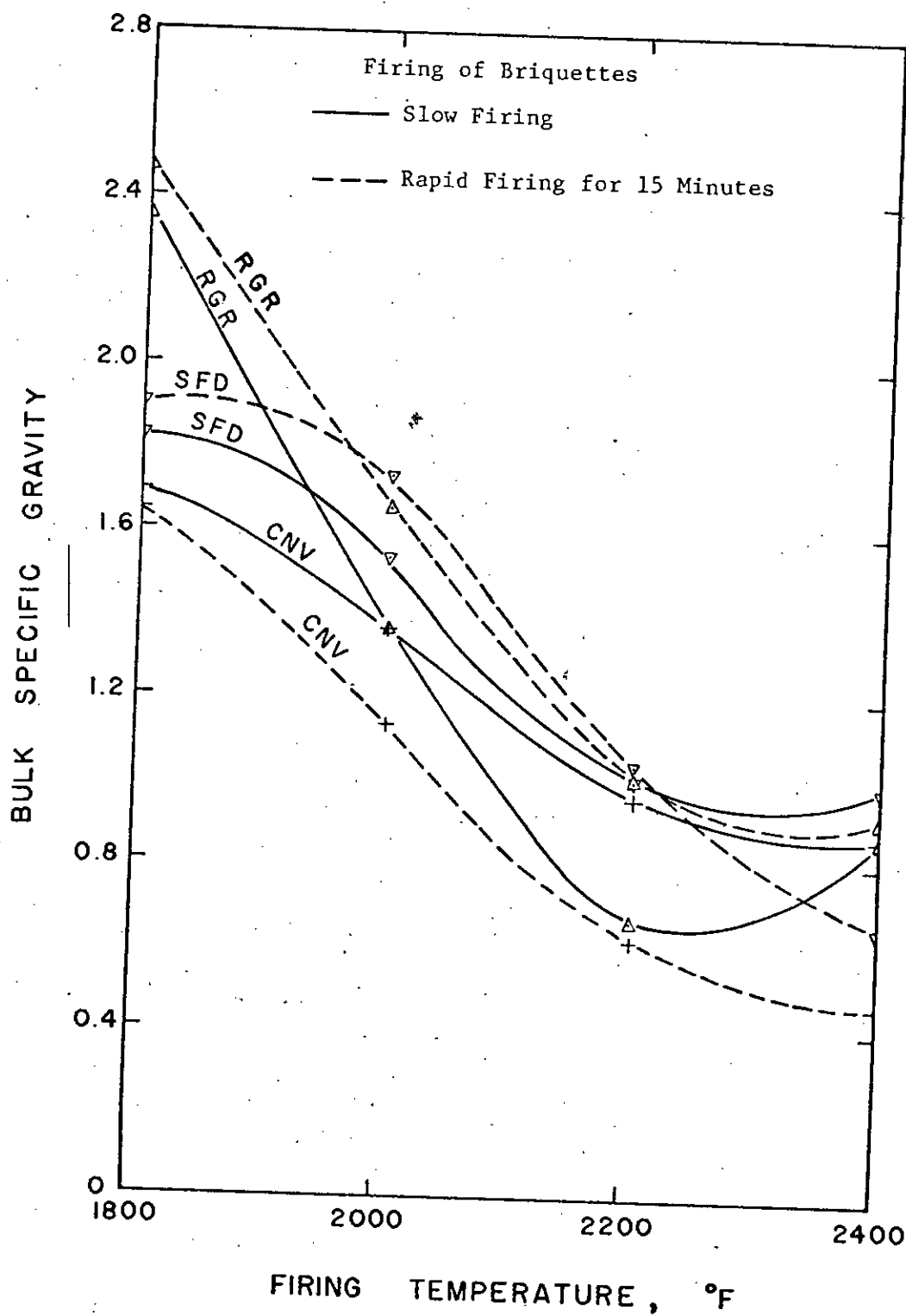


Fig. 15. Effect of Firing Rapidity on Bloating for Samples RGR, SFD, and CNV.

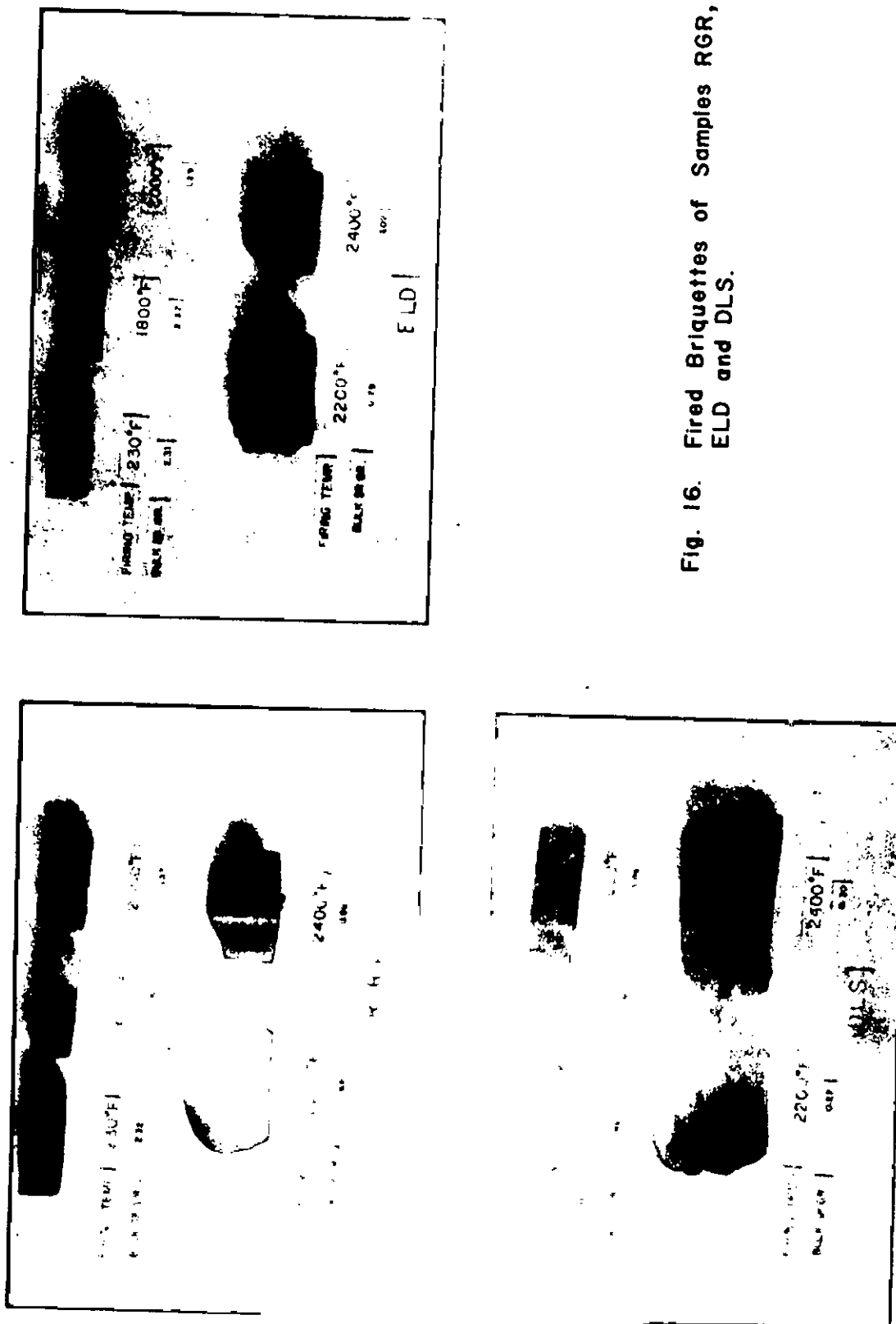


Fig. 16. Fired Briquettes of Samples RGR, ELD and DLS.

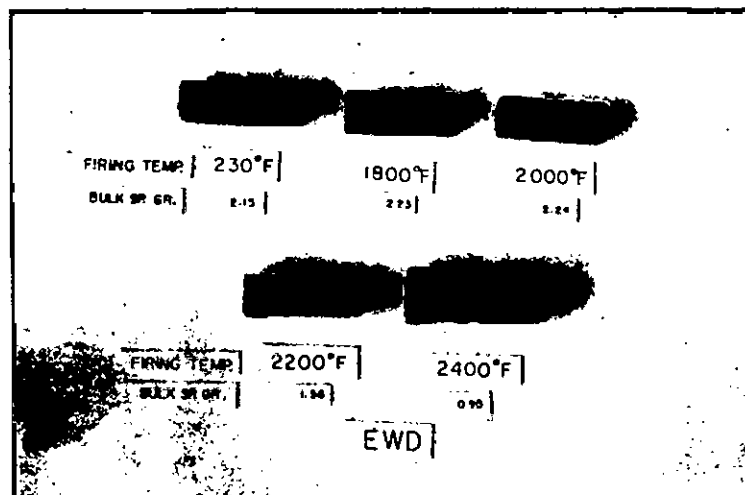
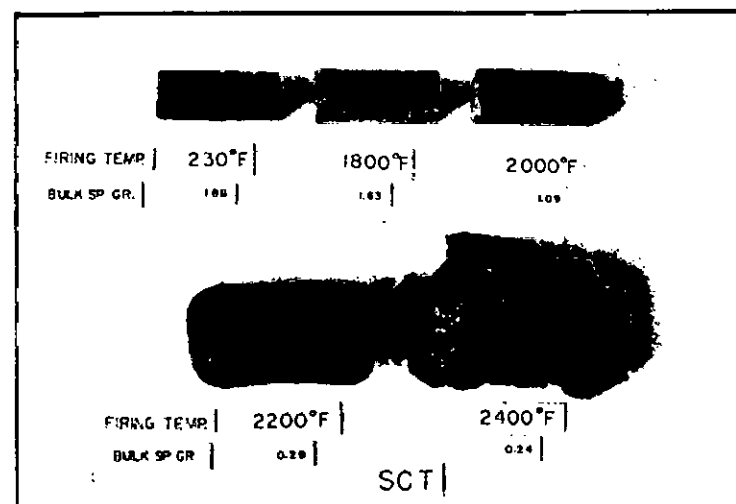
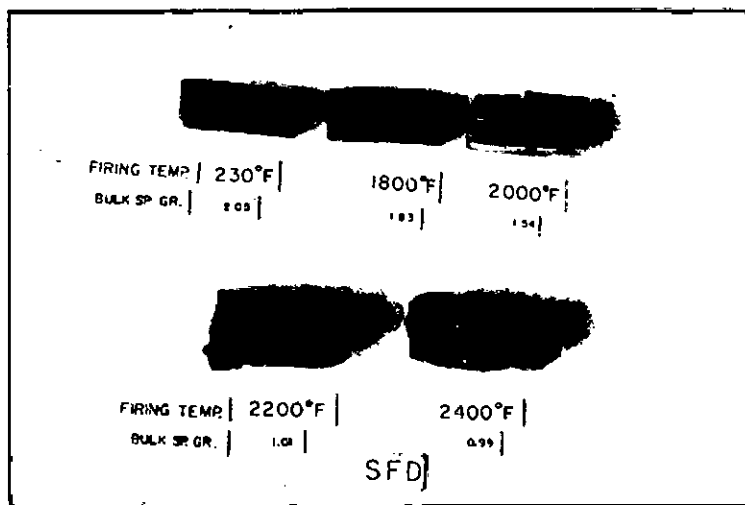


Fig. 17. Fired Briquettes of Samples SFD, SCT and EWD.

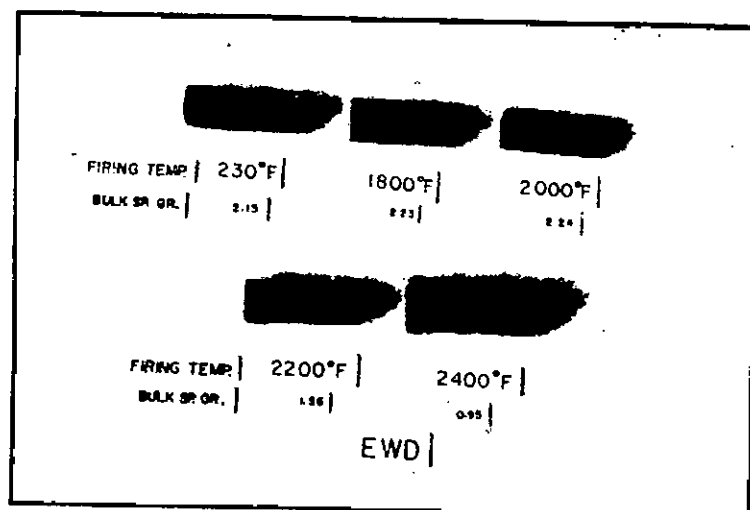
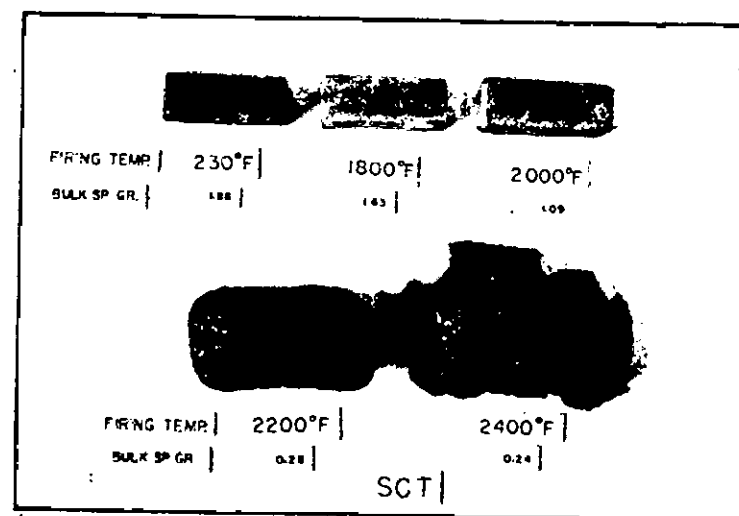
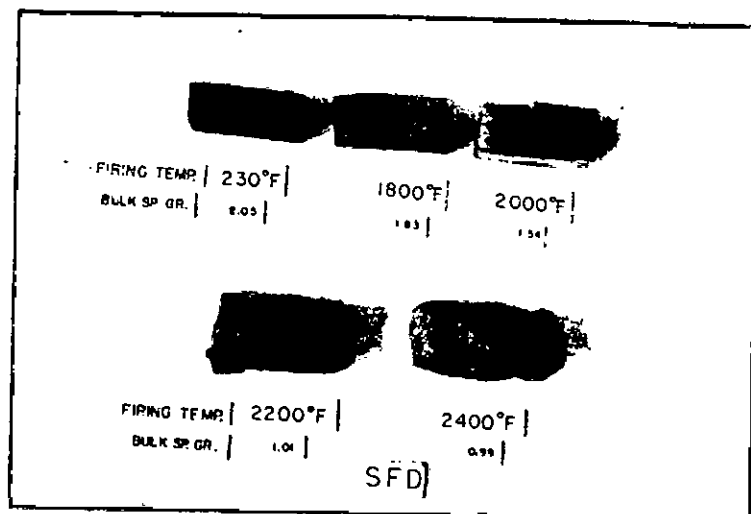


Fig. 17. Fired Briquettes of Samples SFD, SCT and EWD.

temperatures higher than those required for maximum bloating under slow firing.

The effect of firing time, 8 minutes and 15 minutes, on bulk specific gravity of different samples are shown in Table VI and Fig. 18. In general, no significant differences in bloating characteristics was noticed with a change in firing time from 8 minutes to 15 minutes. This indicated that the major part of bloating in each sample took place within first eight minutes or even less. It was first thought that a longer firing time may cause a higher bloating at a lower temperature and at a higher temperature this increase in firing time might cause a lowering of viscosity which would result in the escape of entrapped gases and sometimes in greater fusion of the material. However, over the temperature and time range studied, no such phenomenon was noticed.

During Phase II of this study, the change in bulk specific gravity with firing temperature of sample RGR and DLS was studied by firing crushed (1/4-3/4 in.) particles of these samples. Figure 19 illustrates the difference in bulk specific gravity of remolded briquettes and crushed particles at different firing temperatures under rapid firing for 15 minutes. The bulk specific gravity of the fired product was lower for remolded samples than that for crushed samples. Moreover, as discussed in Chapter II, the bloating of smaller particles is, in general, higher than the bloating of larger particles under

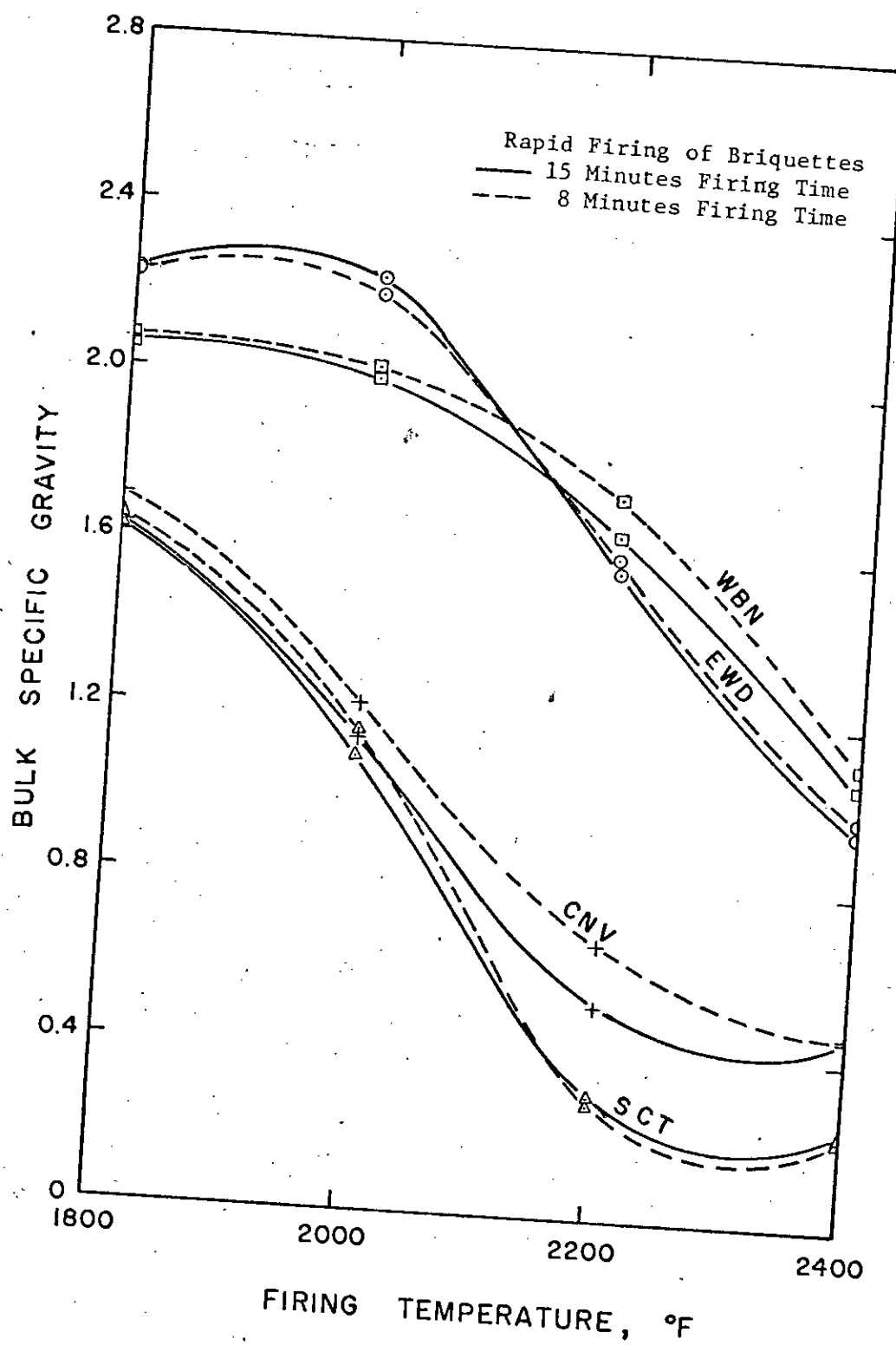


Fig. 18. Effect of Firing Time on Bulk Specific Gravity for Samples SCT, WBN, EWD, and CNV.

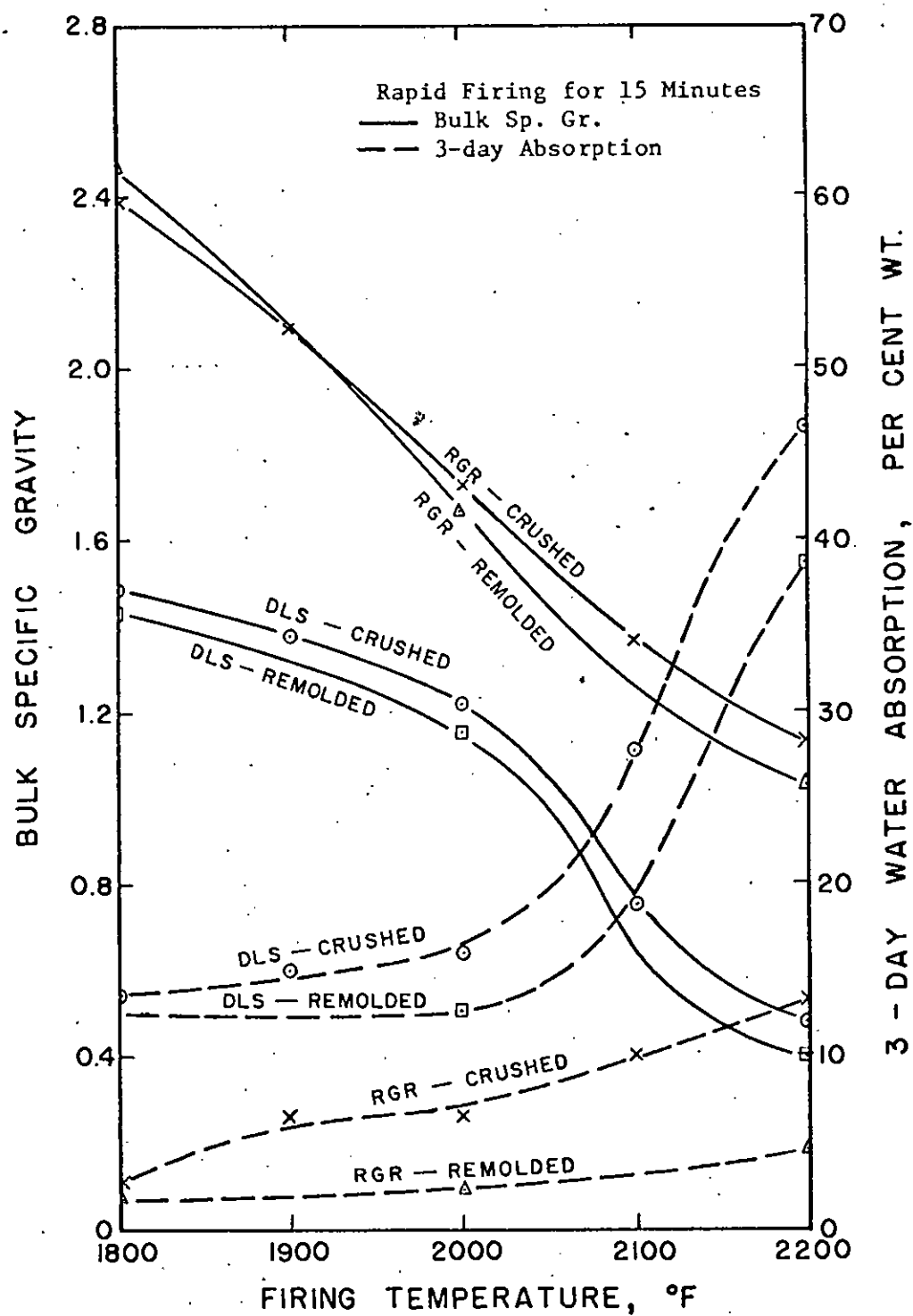


Fig. 19. Effect of Remolding on Bloating and Absorption for Samples RGR and DLS at Different Firing Temperatures.

same conditions of firing. Therefore, the difference in bloating characteristics of remolded and crushed particles would be even more for the same particle size. This increase in bloating with remolding of samples can be attributed to the thorough mixing of gas-producing minerals and fluxing agents in remolded samples.

6.2.2. Absorption. Table VI and Figs. 20 and 21 show the water absorption of different samples at different firing temperatures under both slow and rapid firing of briquettes. Considerable variation was noticed in the absorption characteristics of each sample with firing temperature. This was primarily due to the rapid changes in the internal structure and mineral properties of the material. From a partly or completely dehydroxylated material at 1200°F the sample changed first to a previtrified and partly bloated cellular material at about 1800°F, then to an over-expanded material with interconnected cells at about 2200°F, and finally collapsed with fusion at about 2400°F.

In general, all samples showed a decrease in absorption with a change in firing temperature from 1200 to 1800°F. For samples DLS and SFD, the per cent absorption was higher at 1800°F than that at 1200°F because of the considerable expansion within this temperature interval. Within the temperature range of 1800 to 2200°F, each sample that showed a significant increase in bloating also indicated an increase in

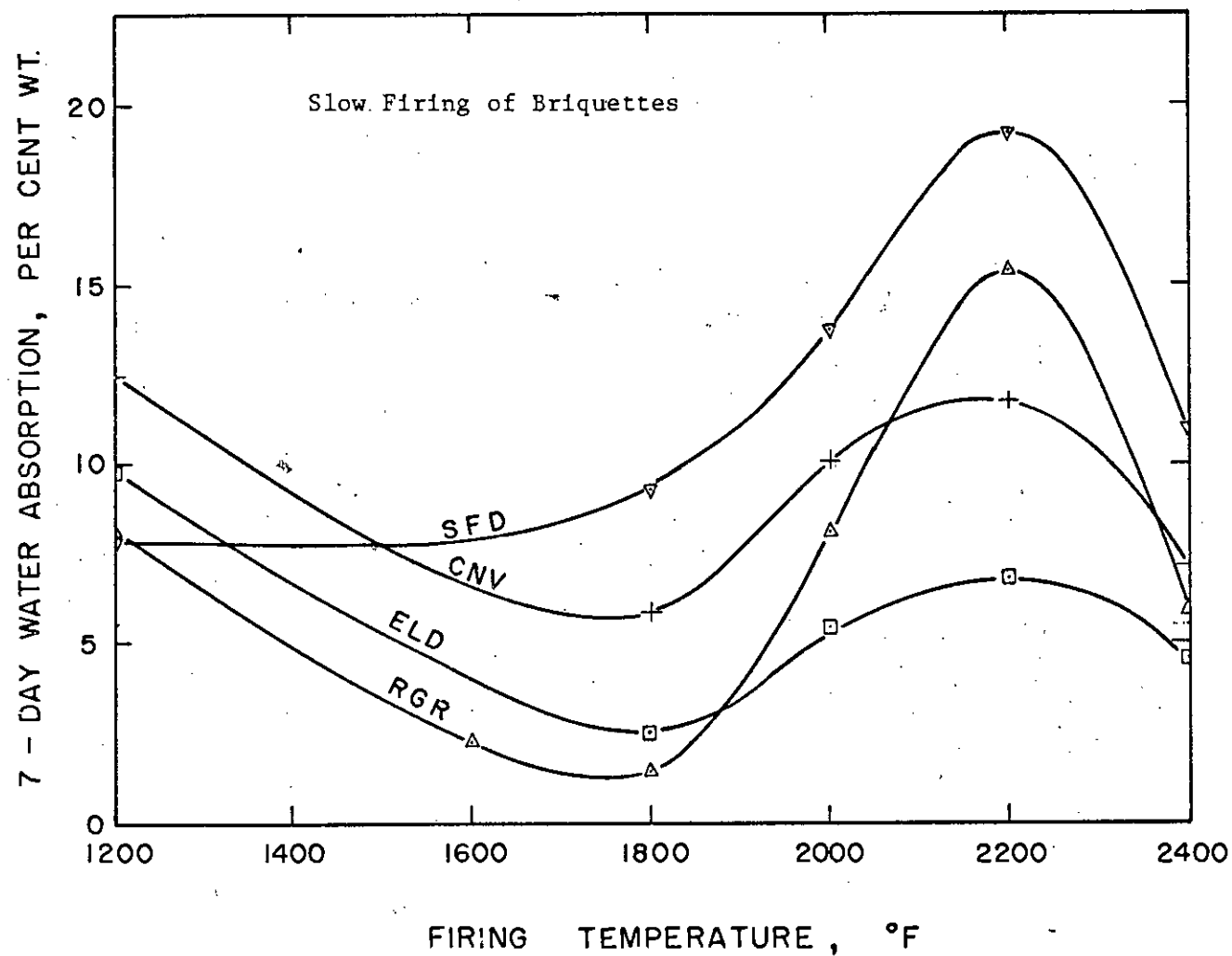


Fig. 20. Effect of Firing Temperature on Absorption for Samples RGR, ELD, CNV, and SFD.

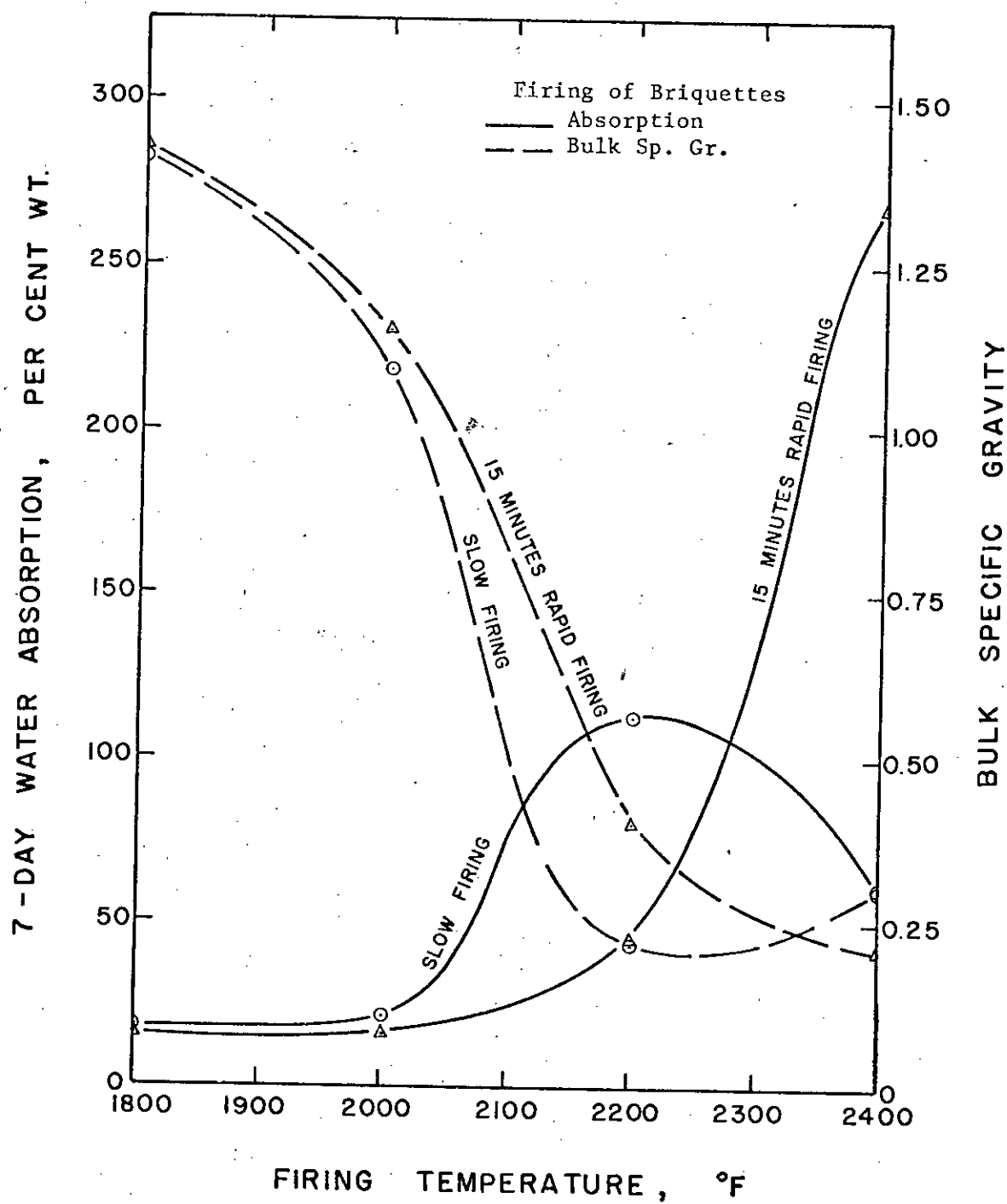


Fig. 21. Effect of Firing Rapidity on Absorption for Sample DLS.

absorption. Deviation from this general behavior was noticed in samples EWD and WBN which showed a decrease in absorption at 2200°F, primarily because of the development of an impermeable glassy phase at this temperature.

At 2400°F each sample fired under slow firing showed a decrease in water absorption, with the development of a partly collapsed cellular structure. The maximum bloating of samples DLS, SFD, CNV, EWD, and SCT at 2400°F under rapid firing caused their maximum absorptions at this temperature. In general, absorption in each sample showed an increase with a decrease in bulk specific gravity. However, no precise relationship was found between these two properties, owing to the fact that bulk specific gravity is a function of both permeable and impermeable pores in the material, whereas absorption is a function of the former only. The formation of impermeable pores with the development of glassy phase caused a decrease in water absorption at higher temperature in some samples. The presence of excess amounts of gas-producing minerals in a material can cause a highly pervious mass even at its fusion temperature.

No significant change was observed in the absorption characteristic of any sample with a change in firing time from 8 minutes to 15 minutes (Table VI and Fig. 22).

The absorption study of briquettes and crushed materials showed that the changes in absorption of each type with firing

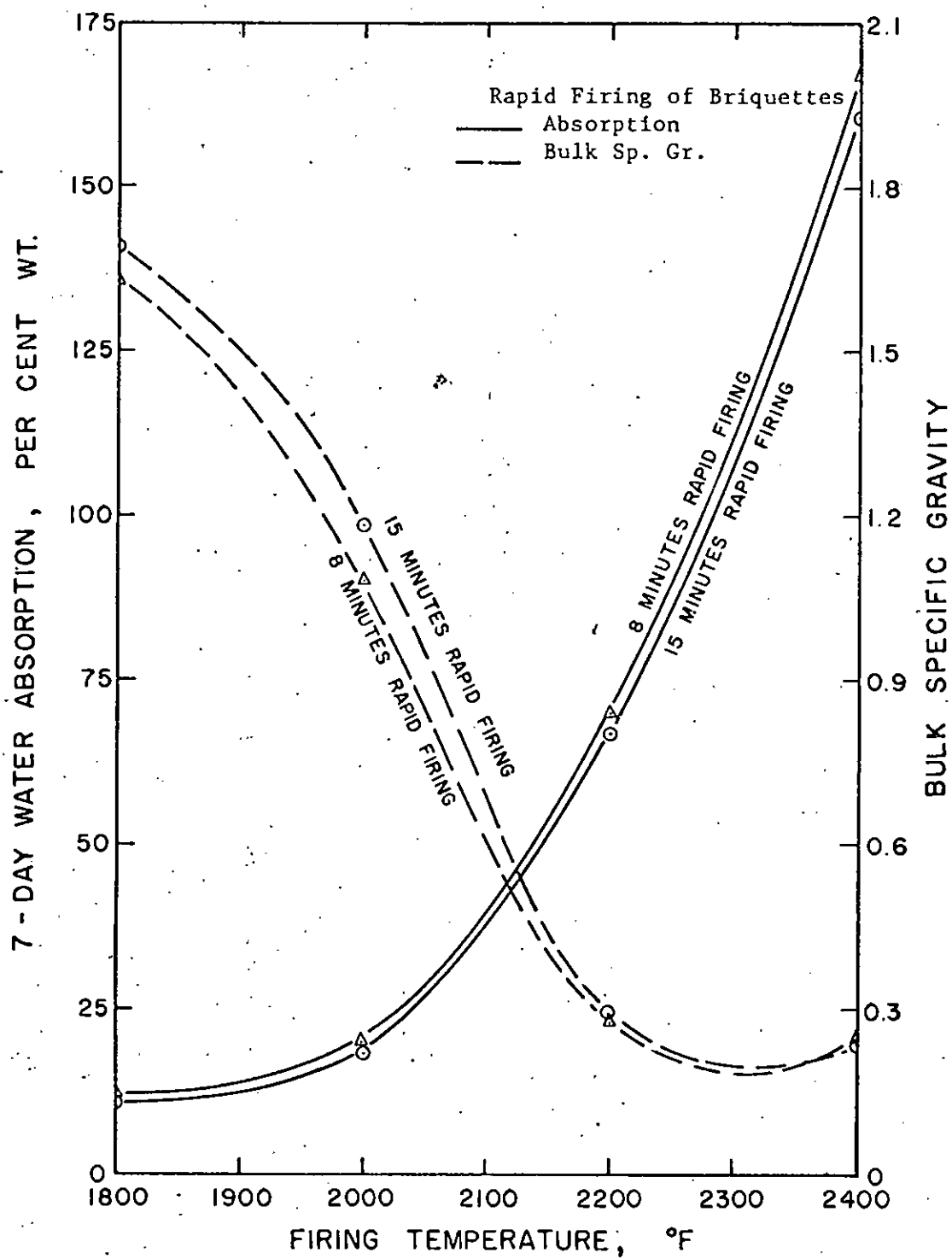


Fig. 22. Effect of Firing Time on Absorption for Sample SCT.

temperature were identical, but the total absorption of briquettes was lower than that of crushed materials (Fig. 19). It is interesting to note that for any given firing temperature, the bloating of a remolded sample was higher than that of a crushed sample, while the absorption was lower. This characteristic behavior of remolded samples may be attributed to the development of better impervious cellular structures in these materials with remolding.

Figures 23 to 32 show the absorption-time curves of different samples under slow and rapid firing. Each sample fired at 1200°F showed complete absorption within the first two hours. This absorption continued for several weeks in samples fired at or above 1800°F. Figures 33 and 34 illustrate 2-hour, 24-hour, 3-day, 7-day, and 14-day absorptions as per cents of 28-day absorption for samples RGR and DLS at firing temperatures ranging from 1800 to 2200°F at 100°F intervals under rapid firing for 15 minutes. In general, each sample absorbed water at a higher rate in the beginning, with about 40 to 50 per cent of the total absorption in 28 days taking place within the first 24 hours. This percentage was little higher for samples fired at 1800°F which may be attributed to their incomplete burning at this temperature. It is also evident from Figs. 33 and 34 that the absorption in any particular period as a per cent of 28 day absorption does not show any appreciable change with firing temperature above 1900°F.

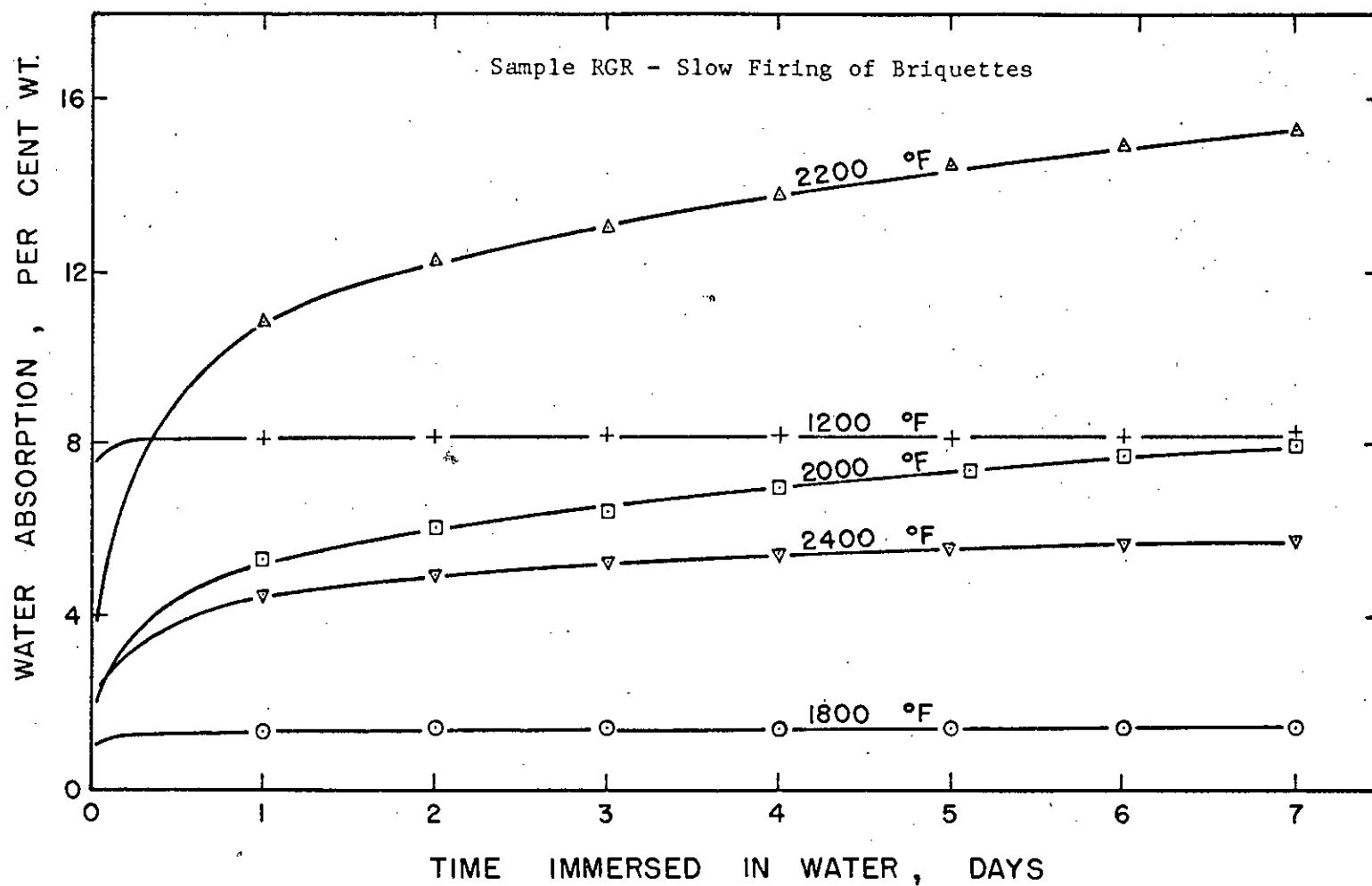


Fig. 23. Absorption-Time Curves for Sample RGR.

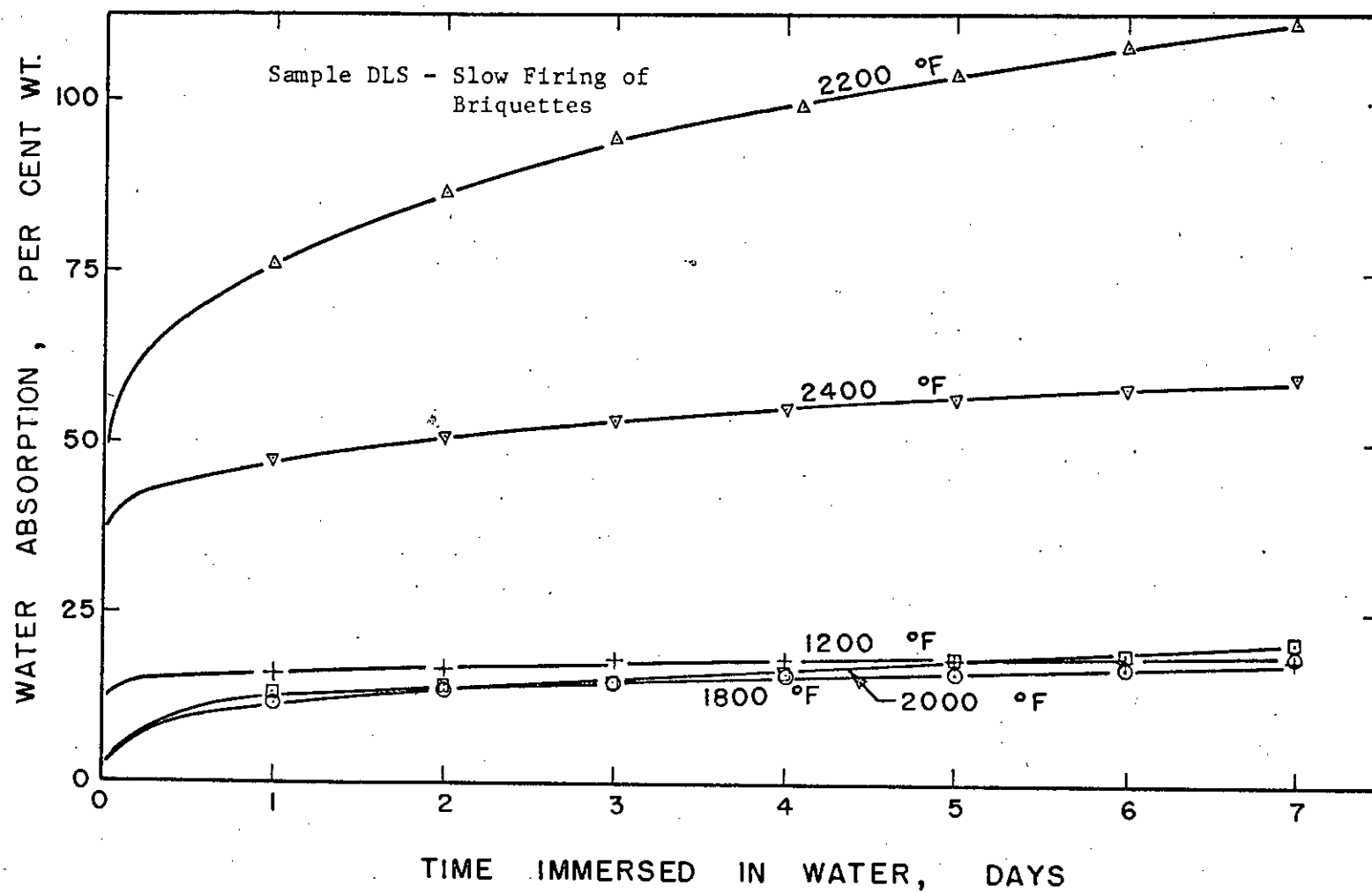


Fig. 24. Absorption-Time Curves for Sample DLS.

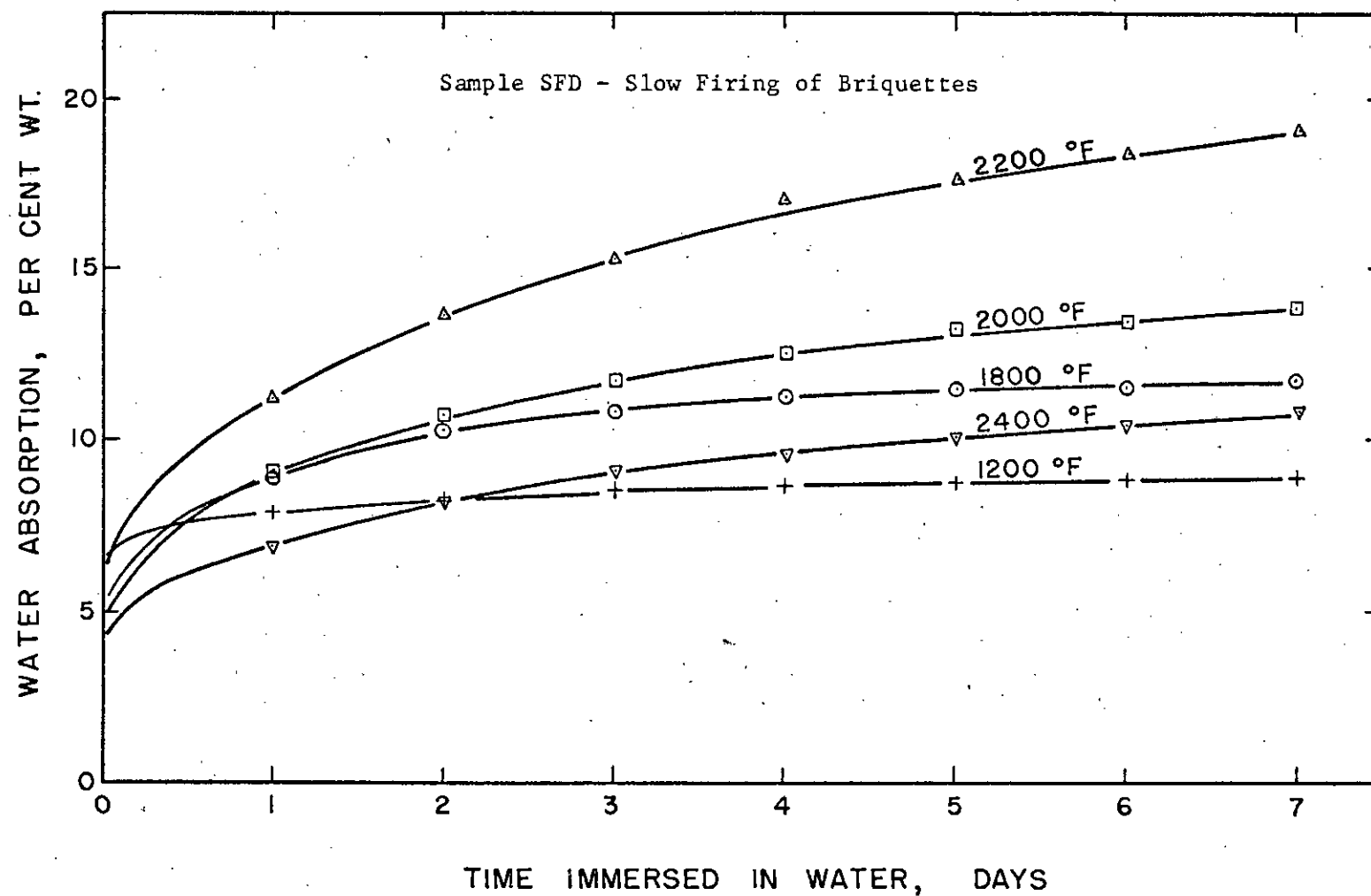


Fig. 25. Absorption-Time Curves for Sample SFD.

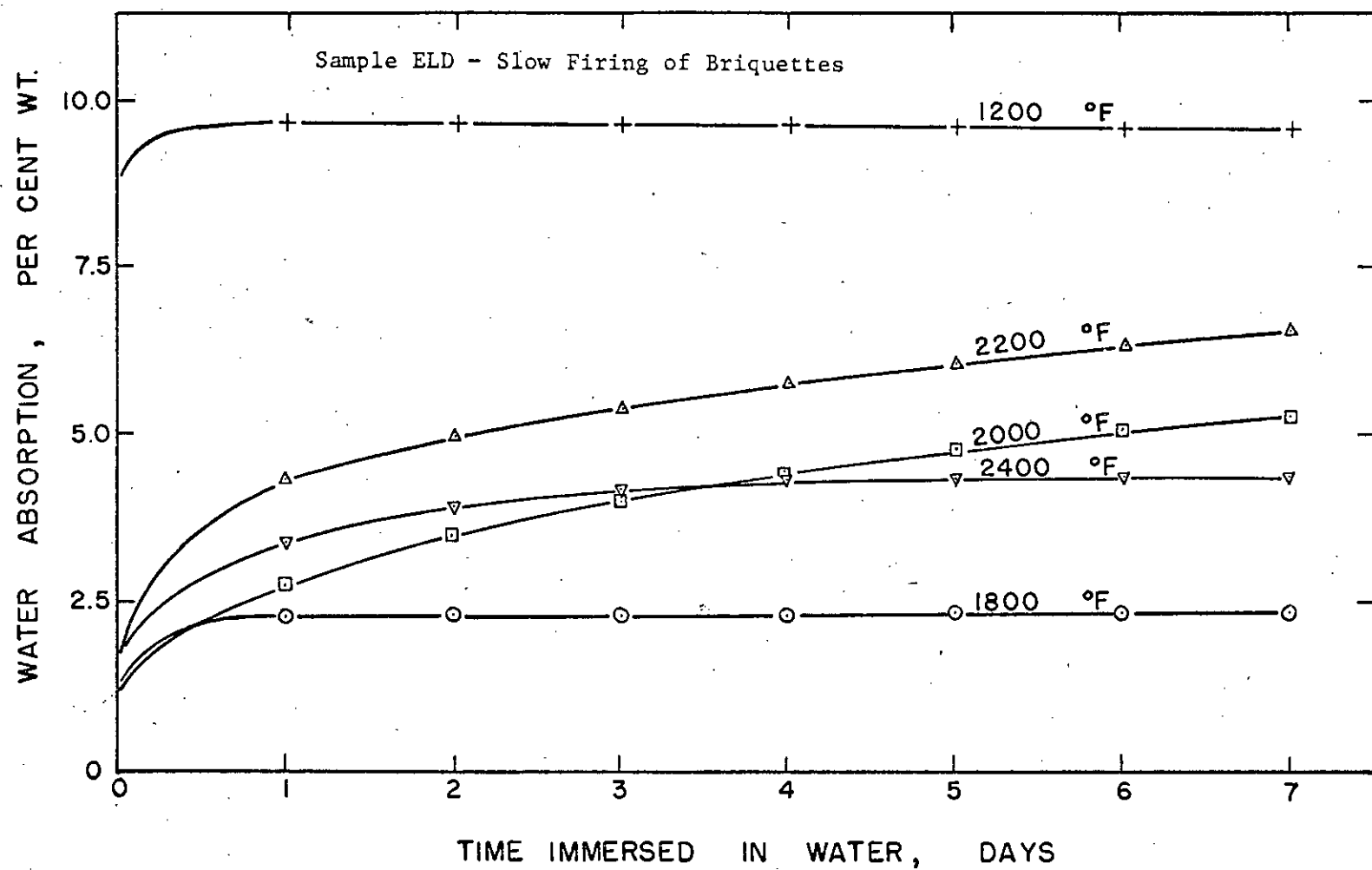


Fig. 26. Absorption-Time Curves for Sample ELD.

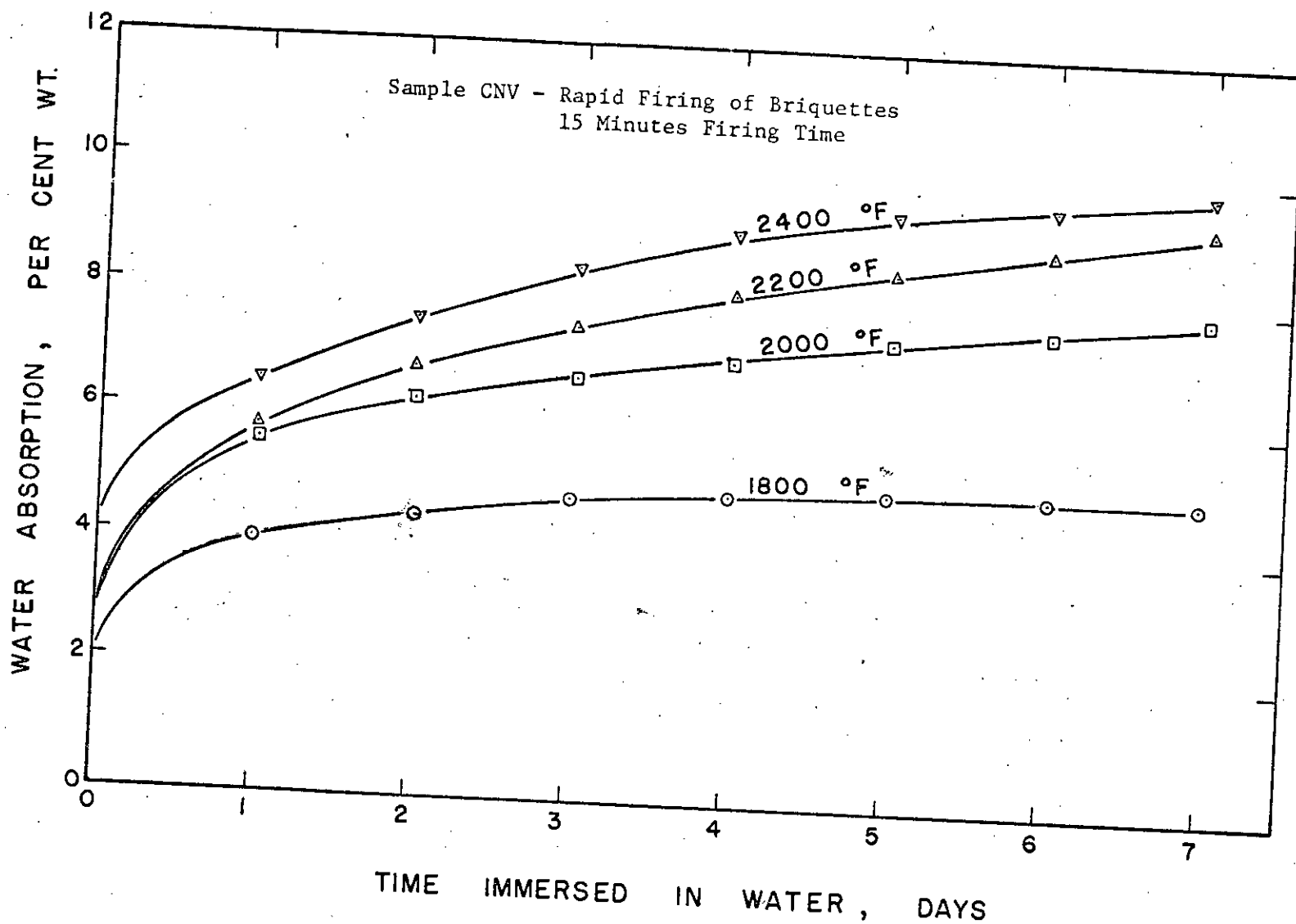


Fig. 27. Absorption-Time Curves for Sample CNV.

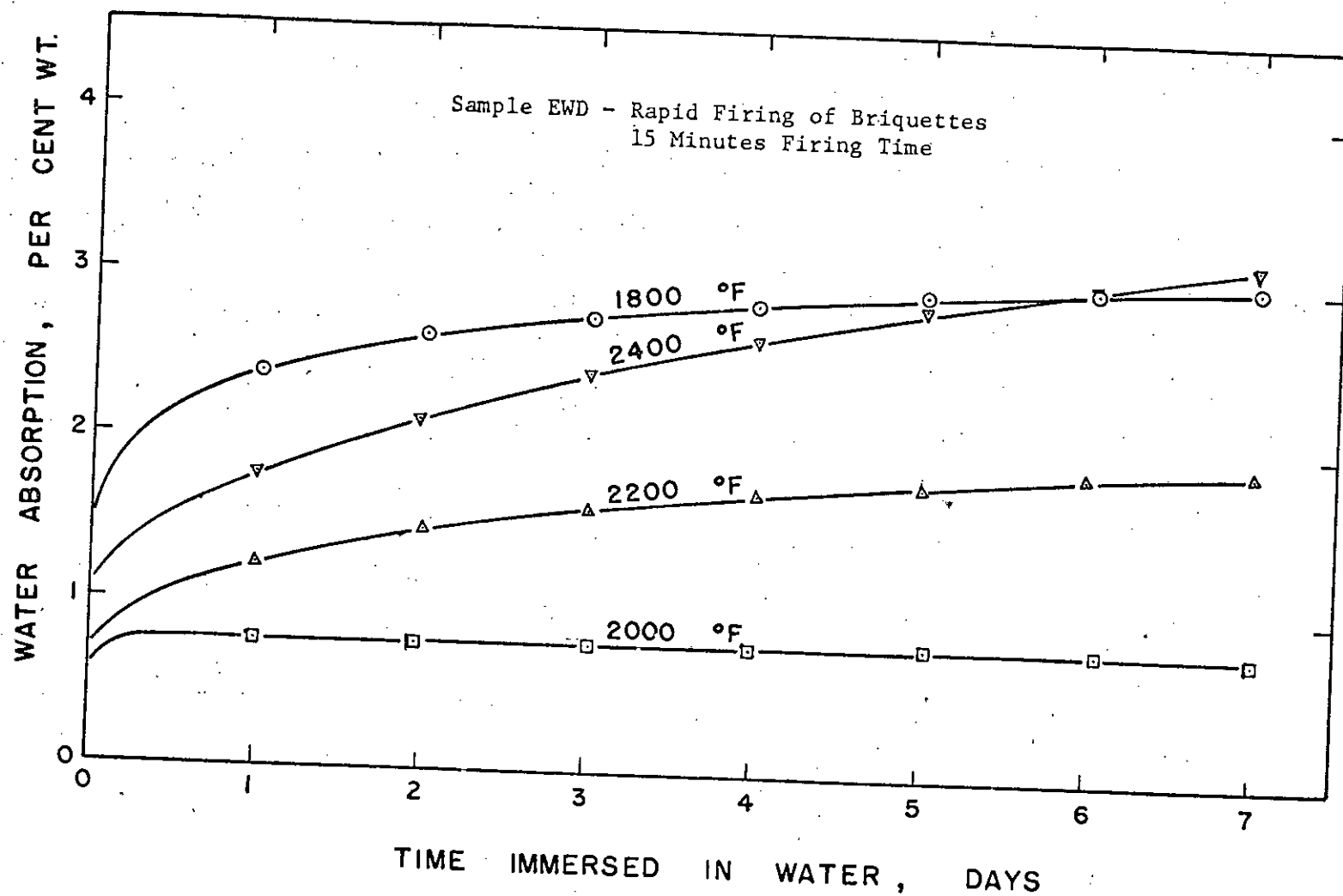


Fig. 28. Absorption-Time Curves for Sample EWD.

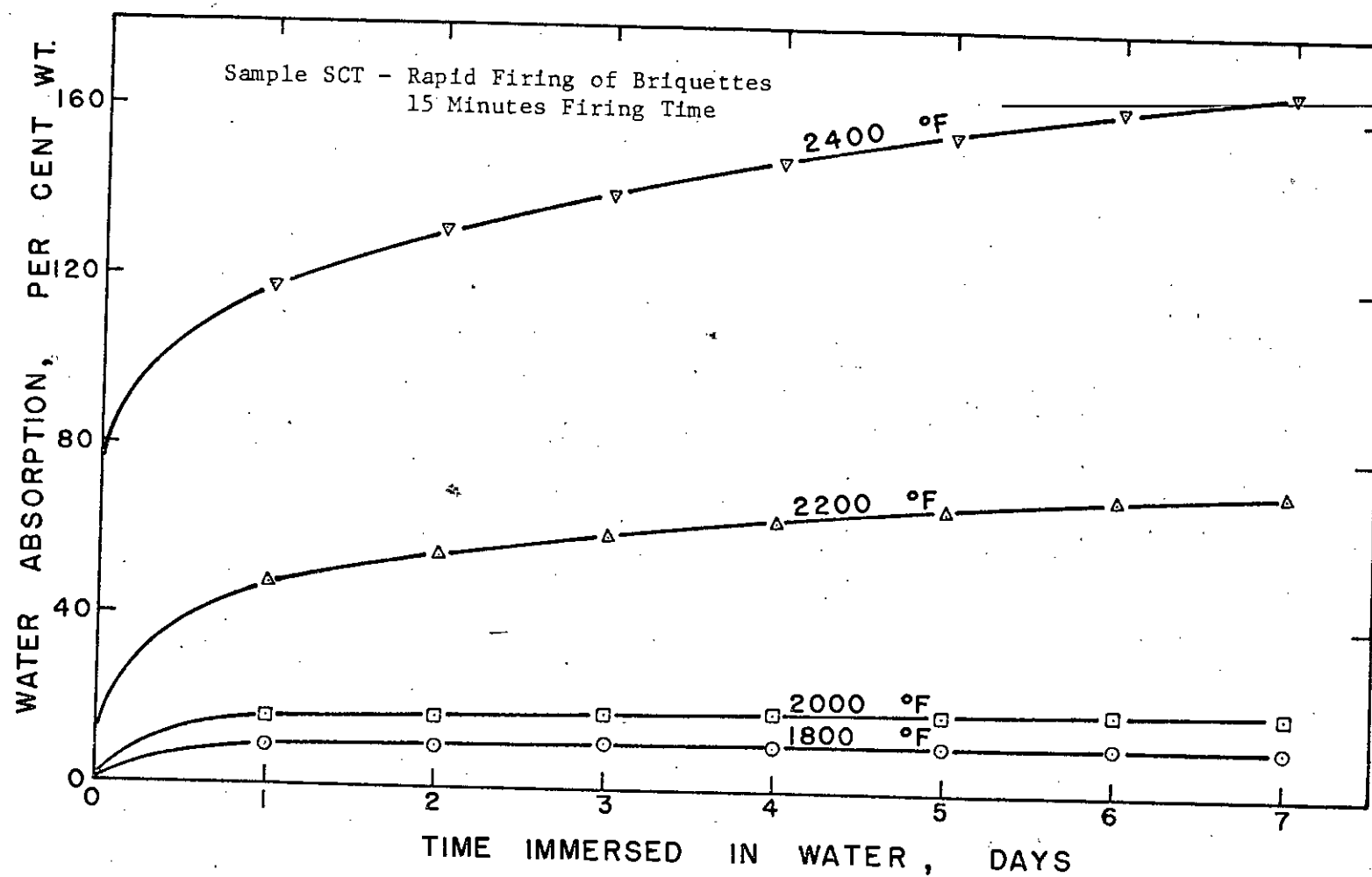


Fig. 29. Absorption-Time Curves for Sample SCT.

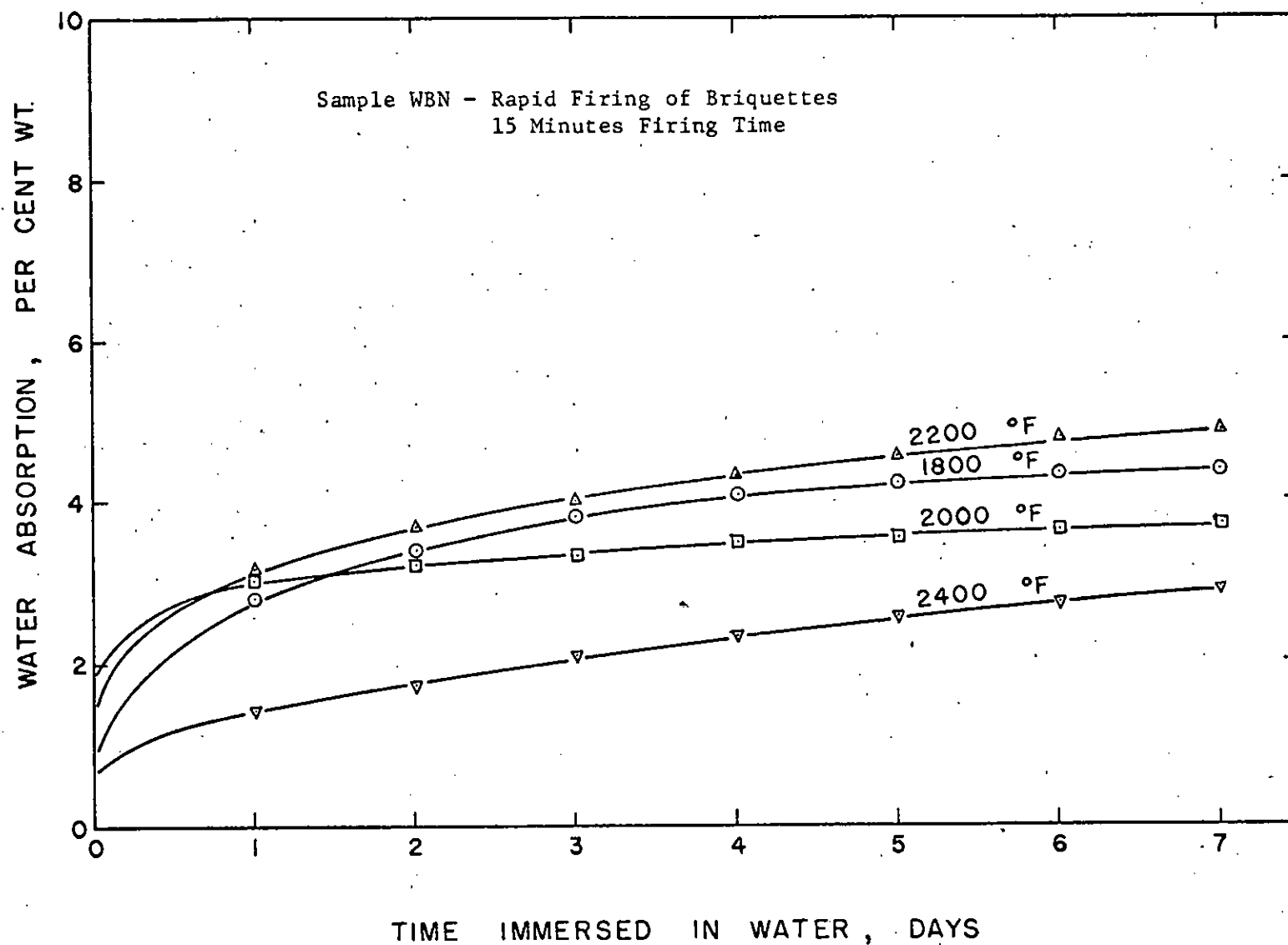


Fig. 30. Absorption-Time Curves for Sample WBN.

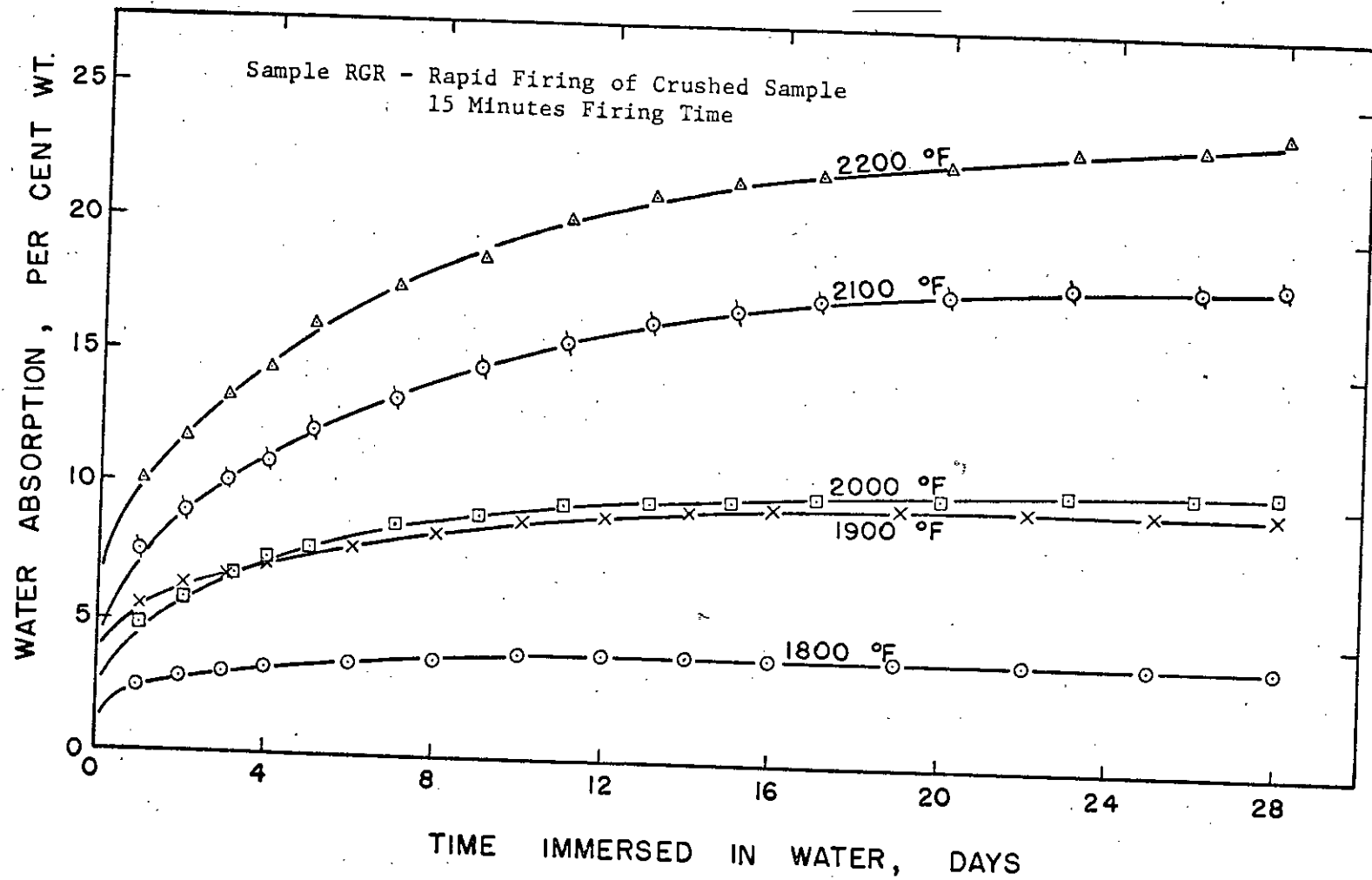


Fig. 31. Absorption-Time Curves for Sample RGR.

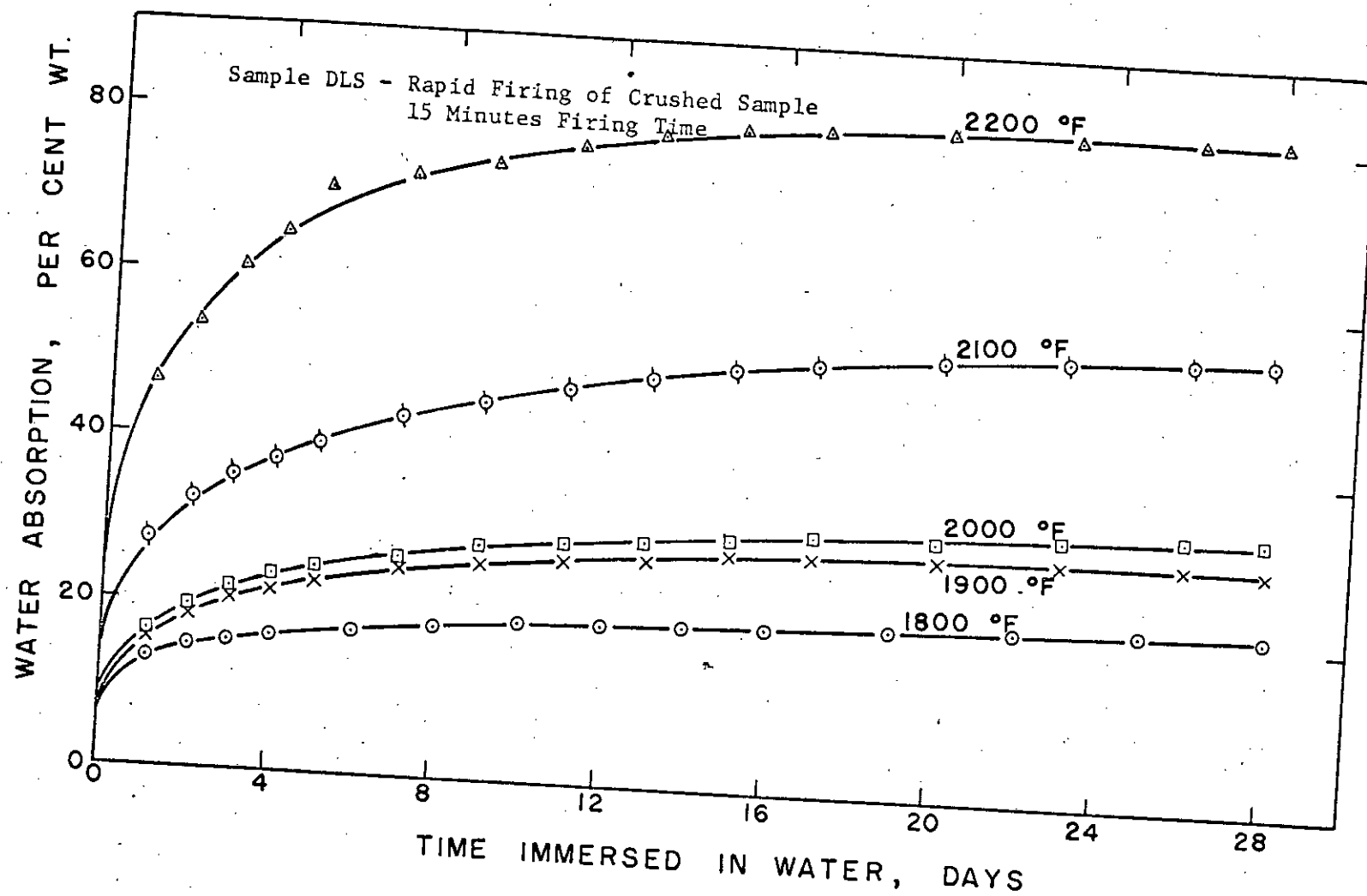


Fig. 32. Absorption-Time Curves for Sample DLS.

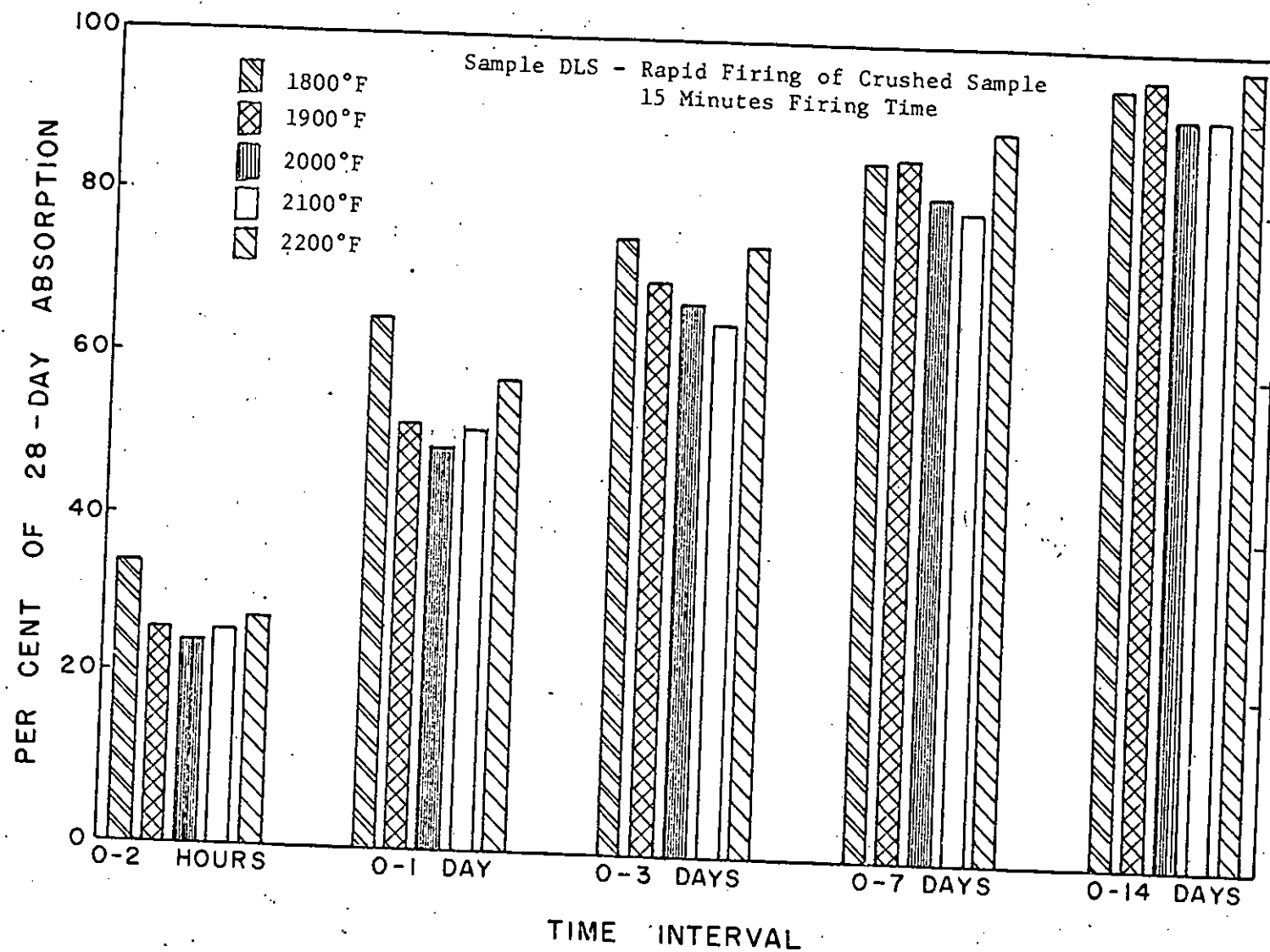


Fig. 33. Effect of Firing Temperature on Rate of Saturation for Sample DLS.

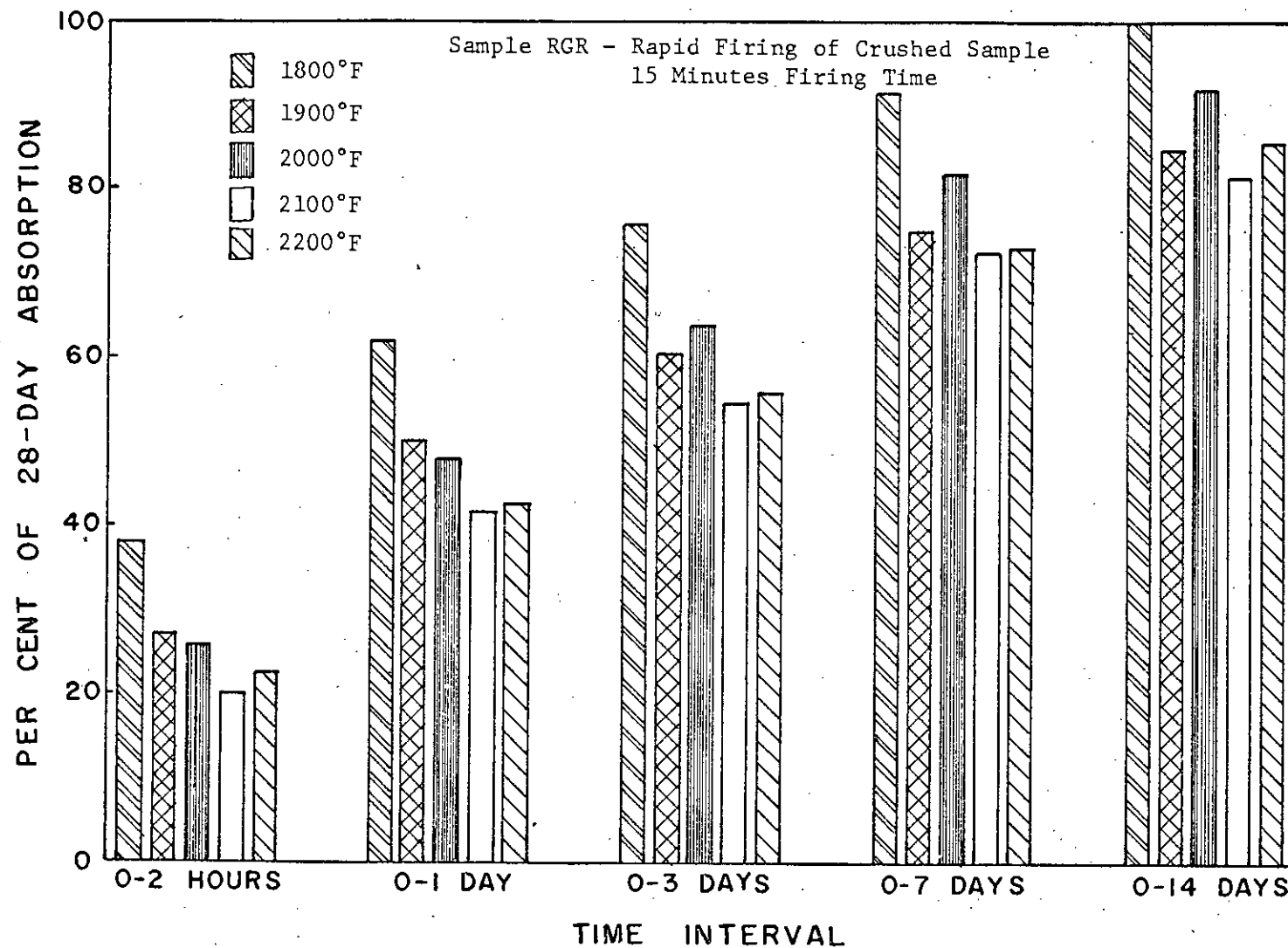


Fig. 34. Effect of Firing Temperature on Rate of Saturation for Sample RGR.

6.2.3. Freeze-Thaw. Tables VII, VIII and Fig. 35 show the freeze-thaw results for samples of crushed DLS and RGR fired at 1800, 2000, and 2200°F under rapid firing for 15 minutes. Table VII illustrates the loss based on per cent passing through the sieve on which all particles were retained before test. The per cent loss for each sample showed a decrease with an increase in firing temperature. At 1800°F the per cent loss was maximum for both the samples. Specimen fractures in most cases, took place along the planes of lamination of shales. Numerous cracks were observed across each particle fired at 1800°F along these planes of weakness before they were subjected to free-thaw. These cracks might have been formed with the development of differential stresses in a particle originating from the sharp temperature gradient in the process of rapid firing and cooling. Differential expansions or contractions of adjacent layers of shale because of their difference in mineral contents could also cause these cracks.

At 2000°F the loss in each sample was greatly reduced with partial fusion of some of its minerals. The reduction was greater for sample RGR. Sample DLS still showed significant loss because of the presence of these interlayer cracks. At 2200°F particles of each sample showed complete vitrification with the elimination of all their laminations. Both samples showed minimum losses at this temperature.

Table VII. Rapid Freeze-Thaw of Synthetic Aggregates Based On Per Cent Weight Loss Through $\frac{1}{2}$ in. Sieve for Group A and $\frac{1}{4}$ in. Sieve for Group B.

Sample	Size Range in.	No. of Particles	Wt. Before Test Gm.	Wt. Loss After 50 Cycles, Gm.*	Wt. Loss* Per Cent
<u>Group A</u>					
DLS					
1800°F	3/4-1/2	100	227.0	117.5	51.9
2000°F	3/4-1/2	100	200.0	43.5	21.8
2200°F	3/4-1/2	100	111.0	6.7	6.0
RGR					
1800°F	3/4-1/2	100	423.5	137.0	32.4
2000°F	3/4-1/2	100	367.5	15.1	4.1
2200°F	3/4-1/2	100	191.0	3.5	1.8
<u>Group B</u>					
DLS					
1800°F	1/2-1/4	200	165.0	42.0	25.5
2000°F	1/2-1/4	200	130.0	7.5	5.7
2200°F	1/2-1/4	200	86.0	1.0	1.1
RGR					
1800°F	1/2-1/4	200	211.0	37.8	17.9
2000°F	1/2-1/4	200	194.0	3.3	1.7
2200°F	1/2-1/4	200	137.0	1.8	1.3

* Weight loss measured through 1/2 inch sieve for Group A and through 1/4 inch for Group B.

Table VIII. Rapid Freeze-Thaw of Synthetic Aggregates Based on Per Cent Weight Loss Through No. 12 Sieve.

Sample	Size Range in.	No. of Particles Before Test	No. of Particles After 50 Cycles	Wt. Before Test Gm.	Wt. Passing No. 12 Sieve Gm.	Per Cent Wt. Passing No. 12 Sieve
<u>Group A</u>						
DLS						
1800°F	3/4-1/2	100	63	227.0	19.2	8.4
2000°F	3/4-1/2	100	84	200.0	21.6	1.3
2200°F	3/4-1/2	100	95	111.0	2.5	2.3
RGR						
1800°F	3/4-1/2	100	79	423.5	10.8	2.5
2000°F	3/4-1/2	100	96	367.5	2.4	0.6
2200°F	3/4-1/2	100	99	191.0	1.0	0.5
<u>Group B</u>						
DLS						
1800°F	1/2-1/4	200	267	165.0	18.3	11.1
2000°F	1/2-1/4	200	218	130.0	4.0	3.1
2200°F	1/2-1/4	200	203	86.0	0.8	0.9
RGR						
1800°F	1/2-1/4	200	285	211.0	7.9	3.7
2000°F	1/2-1/4	200	204	194.0	1.5	0.8
2200°F	1/2-1/4	200	202	137.0	1.2	0.9

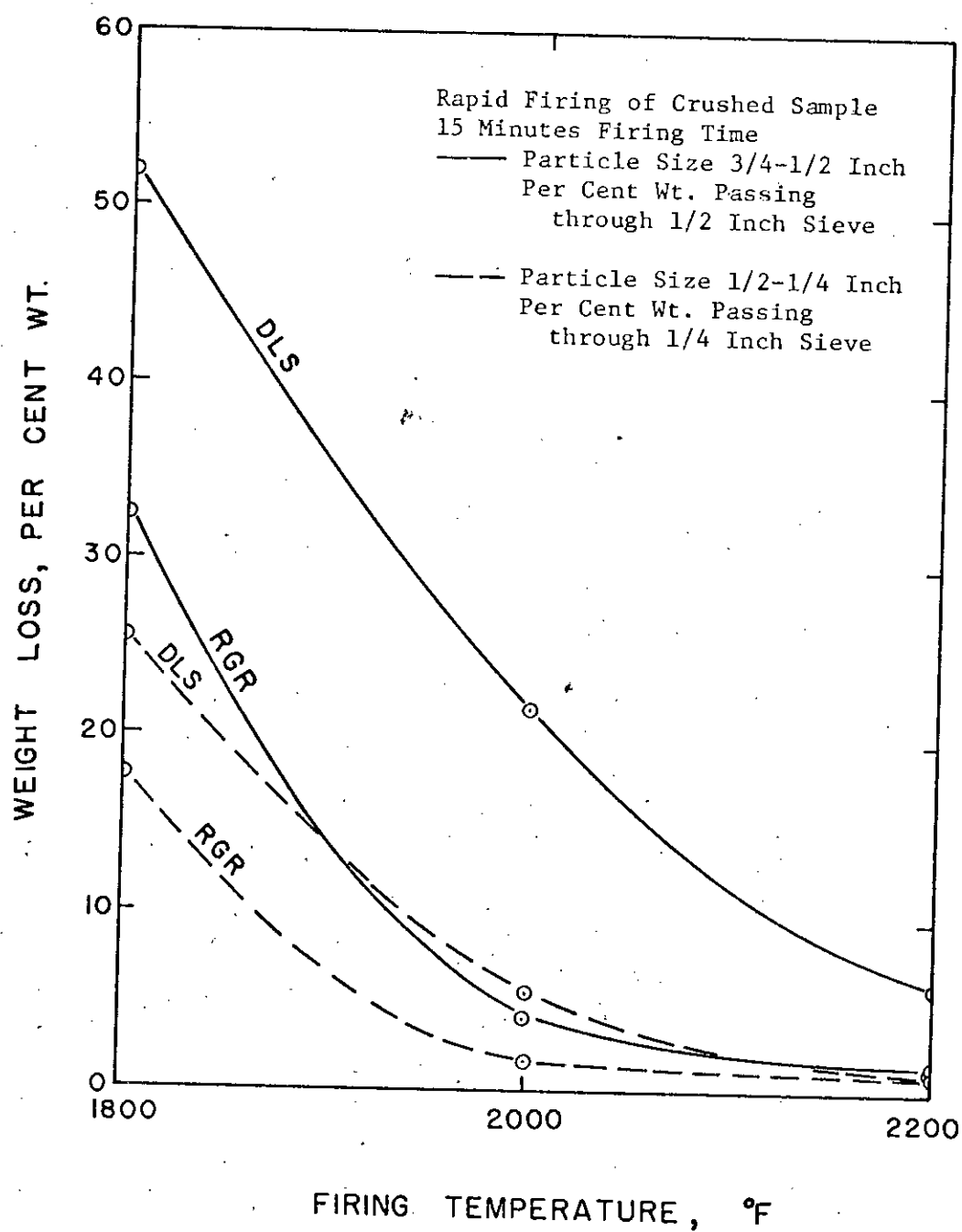


Fig. 35. Effect of Firing Temperature on Resistance to Freeze-Thaw for Samples RGR and DLS.

As all samples were soaked in water for the same period and the rate of saturation of any sample did not show any appreciable variation with firing temperature (Figs. 33 and 34), thus the effect of saturation may be considered to be same for different samples fired at different temperatures. An increase in resistance to freeze-thaw with firing temperatures may also be attributed to an increase in strength of individual grains of each particle with the formation of a more compacted glassy phase at higher temperatures. As freezing of water within a pore causes higher stresses in the grains surrounding it rather than the whole particle, the resistance to freeze-thaw depends primarily on the strength of the former. However, the strength of the whole particle, in general, decreases with an increase in porosity as will be discussed later. Moreover, the pores in a particle at 1800 and 2000°F were irregular with sharp edges as seen in Figs. 10 and 11, but at 2200°F these pores were more or less spherical. Hence, the presence of pores with sharp edges in each sample fired at low temperature caused stress concentrations to develop at these points which finally lead to fracture.

Table VII also shows that with the decrease in particle size, the resistance to freeze-thaw of each sample was appreciably increased. As both larger and smaller particles were prepared by crushing laminated shales, the smaller particles may be expected to be formed by the fracture of weaker planes in larger pieces, thus making the smaller pieces appreciably

stronger. Moreover, smaller pieces might have been vitrified at lower temperatures, where the larger pieces would have only been partially vitrified along their surfaces.

A study of the loss based on per cent passing through No. 12 sieve showed a higher loss for smaller fractions (Table VII). The fraction passing through No. 12 sieve can be considered as the products of surface wearing of individual particle under the action of alternate freezing and thawing. The higher losses in smaller fractions may be attributed to their greater surface areas than the larger fractions of same volumes.

It may be mentioned here that no correlation has yet been found between such a laboratory freeze-thaw test of aggregate particles and the field performance of these aggregates either in concrete or as coverstones subjected to alternate freezing and thawing. However, such tests do provide a means of directly comparing the relative resistances of various samples fired at different temperatures. It may be remarked that failure in concrete under the action of alternate freezing and thawing is primarily caused either by the splitting of individual aggregate particles or by the failure of the bond between cement matrix and aggregate particles rather than the surface wear. The smaller particles at any particular firing temperature would increase the durability of a concrete under such action. As a coverstone where the per cent wearing rather than the fracture of individual particles is considered to be detrimental,

the larger particles are expected to give better performance.

6.2.4. Abrasion. Figure 36 illustrates the results of the modified Los Angeles abrasion test. The percentage losses have been determined by using the fractions passing through No. 4 sieve and No. 12 sieve. The existence of an optimum firing temperature with a minimum per cent wear was noticed for each sample. At 1800°F each sample showed considerable per cent wear which decreased to a minimum at about 2000°F. This increase in resistance even with a decrease in bulk specific gravity can be attributed to the partial vitrification of laminated shales at about 2000°F. At low firing temperatures, the particles tended to split along their planes of lamination, resulting in a very weak material. Again at temperatures above 2100°F, an over expansion of each sample caused a great reduction in its resistance to abrasion, the reduction being higher for sample DLS.

The difference between the per cent losses based on No. 4 and No. 12 sieves indicated the amount of material that was broken because of the brittleness of these particles. It may be noticed in Fig. 35 that this difference was minimum at about 2000°F for each sample. However, this does not necessarily indicate the greater brittleness of the material at 1800°F than at 2000°F. The greater difference at 1800°F was primarily because of the presence of planes of lamination, or planes of weakness, which were welded at about 2000°F with

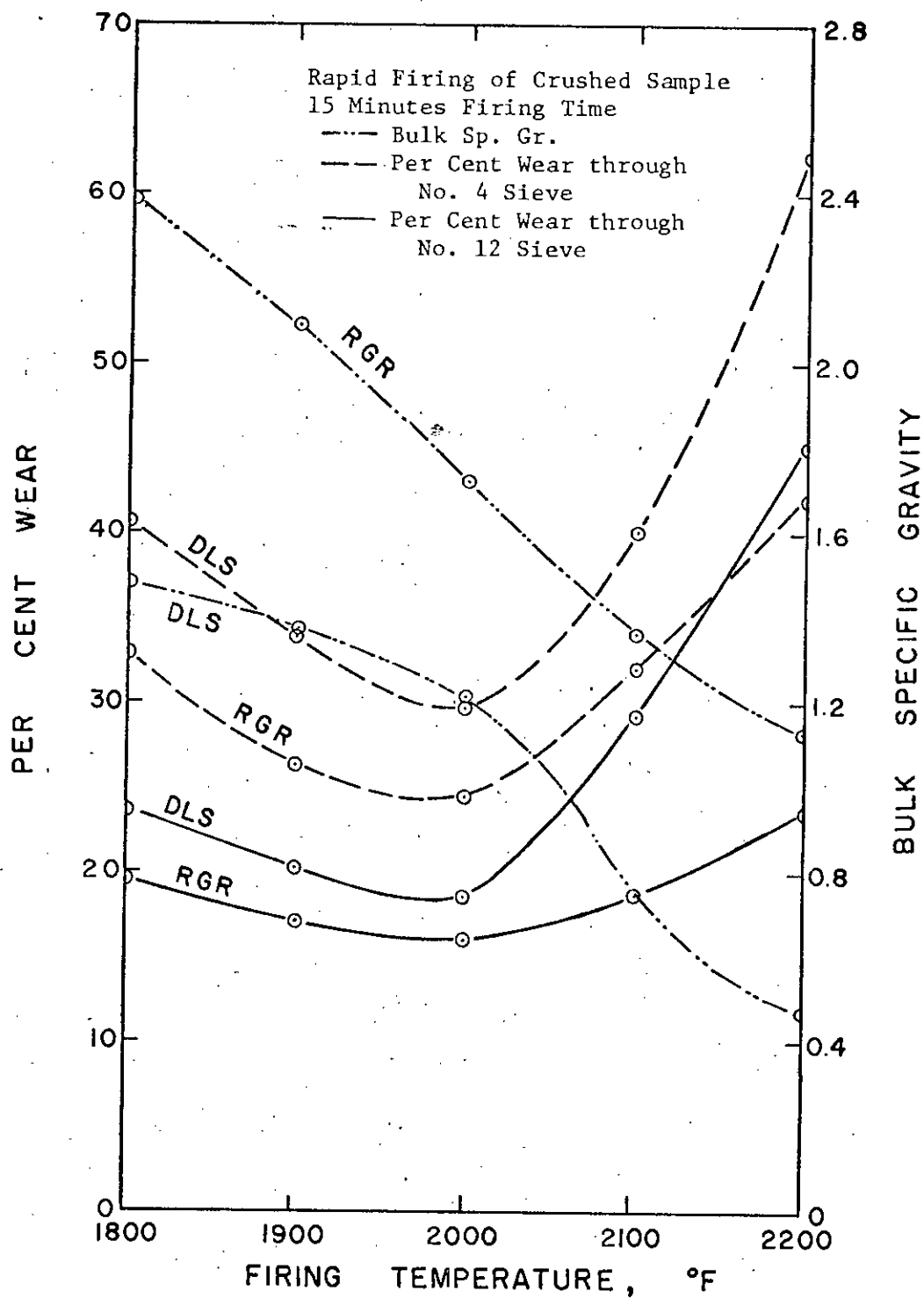


Fig. 36. Effect of Firing Temperature on Per Cent Wear in Modified L.A. Abrasion Test for Samples DLS and RGR.

vittrification of the material. The higher difference at 2200°F than at 2000°F, of course, indicated an increase in brittleness at 2200°F.

It may be mentioned that although the Los Angeles abrasion test shows extremely good correlation with the actual wear of gravel aggregate when used in concrete, the validity of such test for synthetic aggregate is still to be verified. However, the test method used in this work does satisfactorily indicate the change in the resistance of such material against abrasion and attrition with firing temperature.

6.2.5. Potential Reactivity. The results of chemical tests on samples DLS and RGR fired at 1800, 2000, and 2200°F under rapid firing for 15 minutes are shown in Fig. 37. All samples except RGR fired at 2200°F were indicated as non-reactive materials. The solid line in Fig. 36 represents the line of separation between reactive and non-reactive aggregates as proposed by Mielenz and others (33) and finally recommended by ASTM Specification C289-64.

In general, each sample showed an increase in dissolved silica content with firing temperature which was probably caused by the formation of an increased glassy phase at higher temperatures. The formation of high temperature silicate structures, such as spinel, mullite, and cristobalite, the reactions of which with alkali cement are still largely unexplored, may also cause a deleterious effect in concrete. The reduction in alkalinity

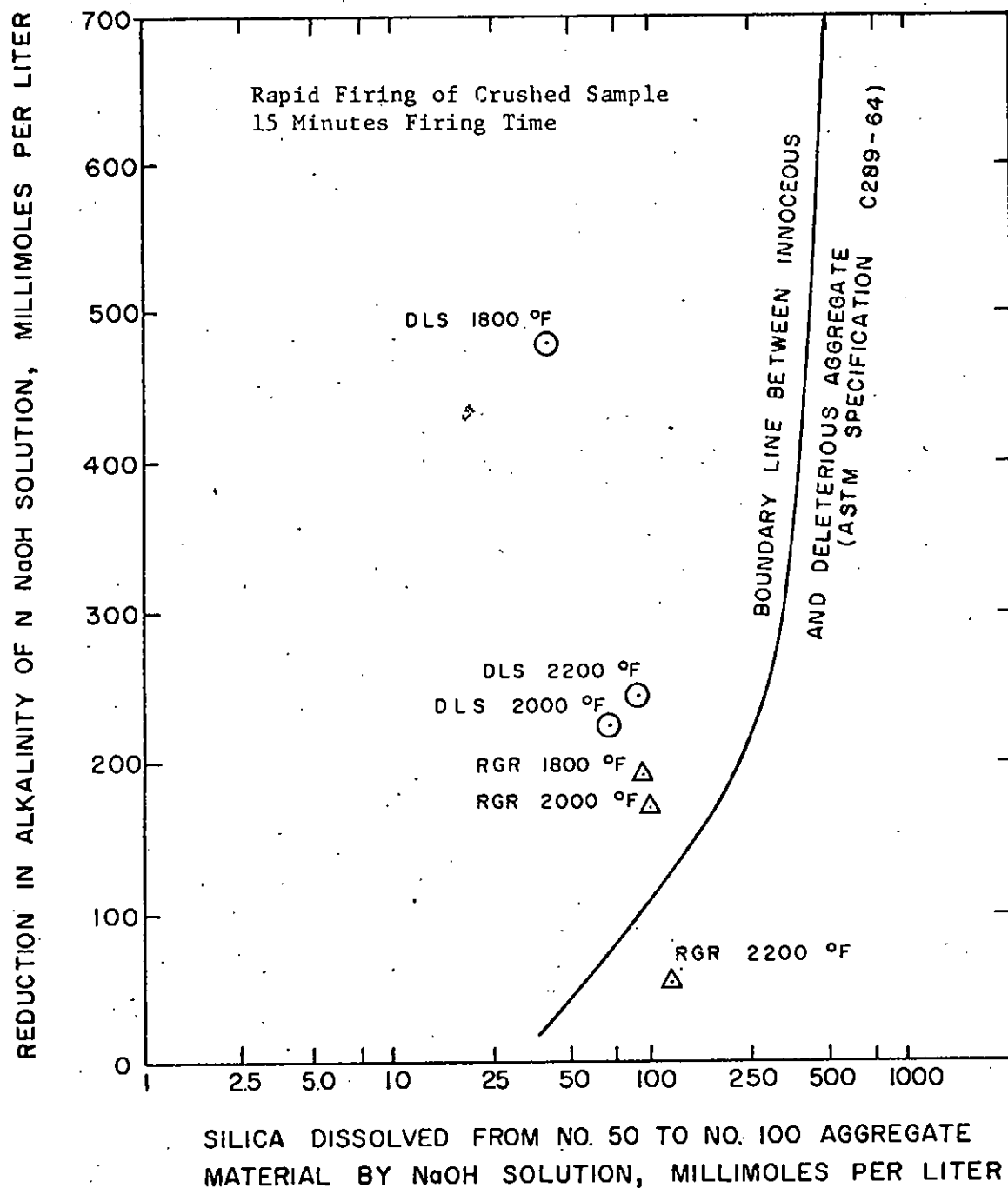


Fig. 37. Results of Potential Reactivity Tests (ASTM Designation C289-64) for Samples DLS and RGR.

in each sample also showed a decrease with firing temperature.

Alkalinity. A study of pH-values of pulverized samples with deionized water indicated high alkalinity of fired products of each sample. The formation of H_2S in sample DLS fired at $1800^{\circ}F$ was detected by its odor which was probably formed by the dissociation of FeS remaining in this underburned sample. Figures 38 and 39 illustrate the change in alkalinity with time for samples DLS and RGR immersed in deionized water. No correlation between firing temperature and pH-value was noticed for any sample. However, each sample fired at different temperatures showed a reduction in alkalinity with time. This was probably because of the slow diffusion of some gases like CO, CO_2 , SO_2 , SO_3 , and H_2S from the pores into water. These gases are believed to be the bloating agents in most clays and shales. The action of these gases on concrete has not yet been studied. However, it is expected that these gases can form some acids causing a poor durability of concrete. The compound FeS remaining in underburned material can cause poor durability of concrete with the formation of sulfates and sulfuric acid in presence of water and air.

It is also known that if the aggregate itself contains appreciable amount of alkalies, as each of these samples did, it can cause expansion by alkali-aggregate reaction with cements containing no alkali. Pike and his colleagues (34) reported that binary sodium and potassium silicate glasses in

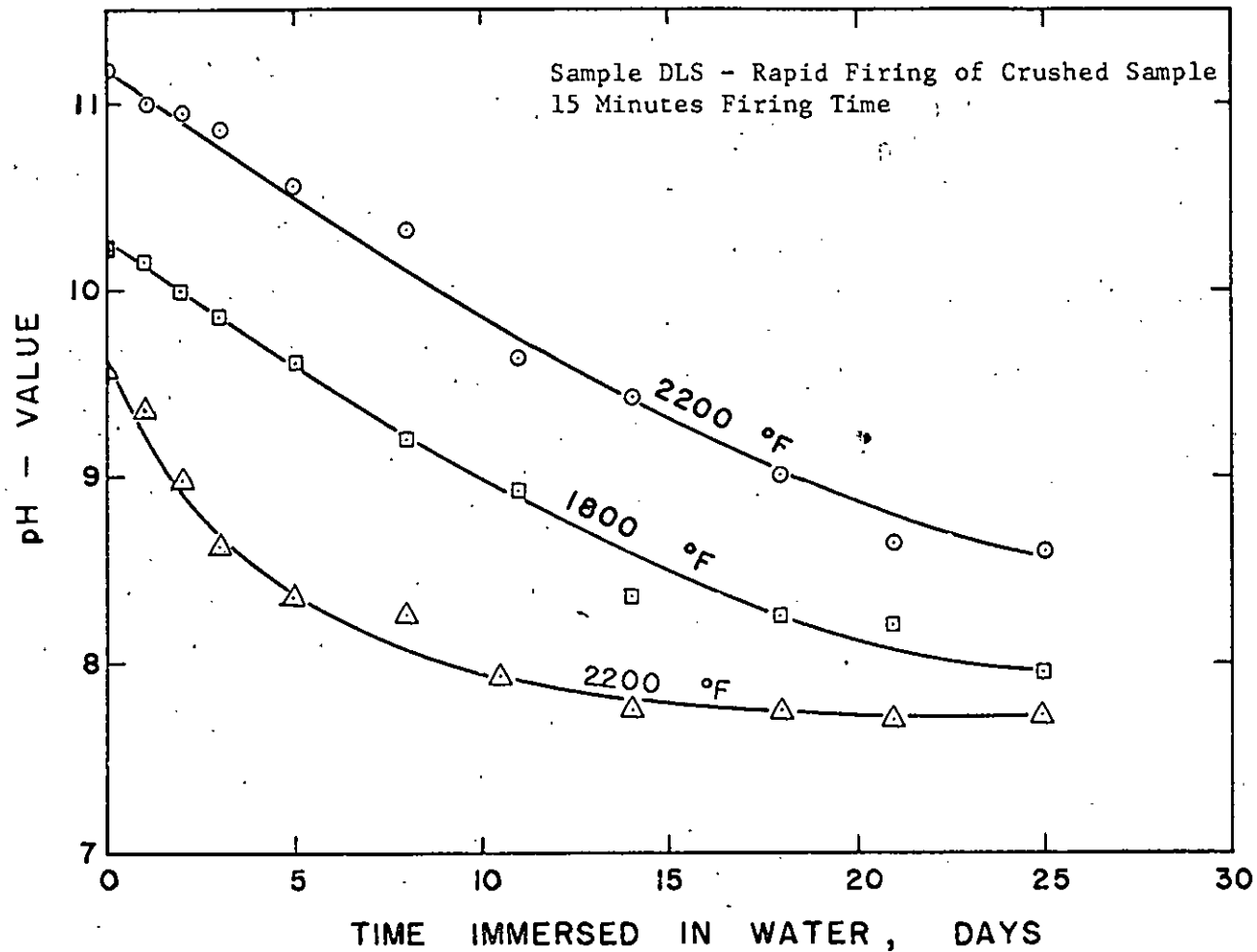


Fig. 38. Change in Alkalinity with Time for Sample DLS Immersed in Deionized Water.

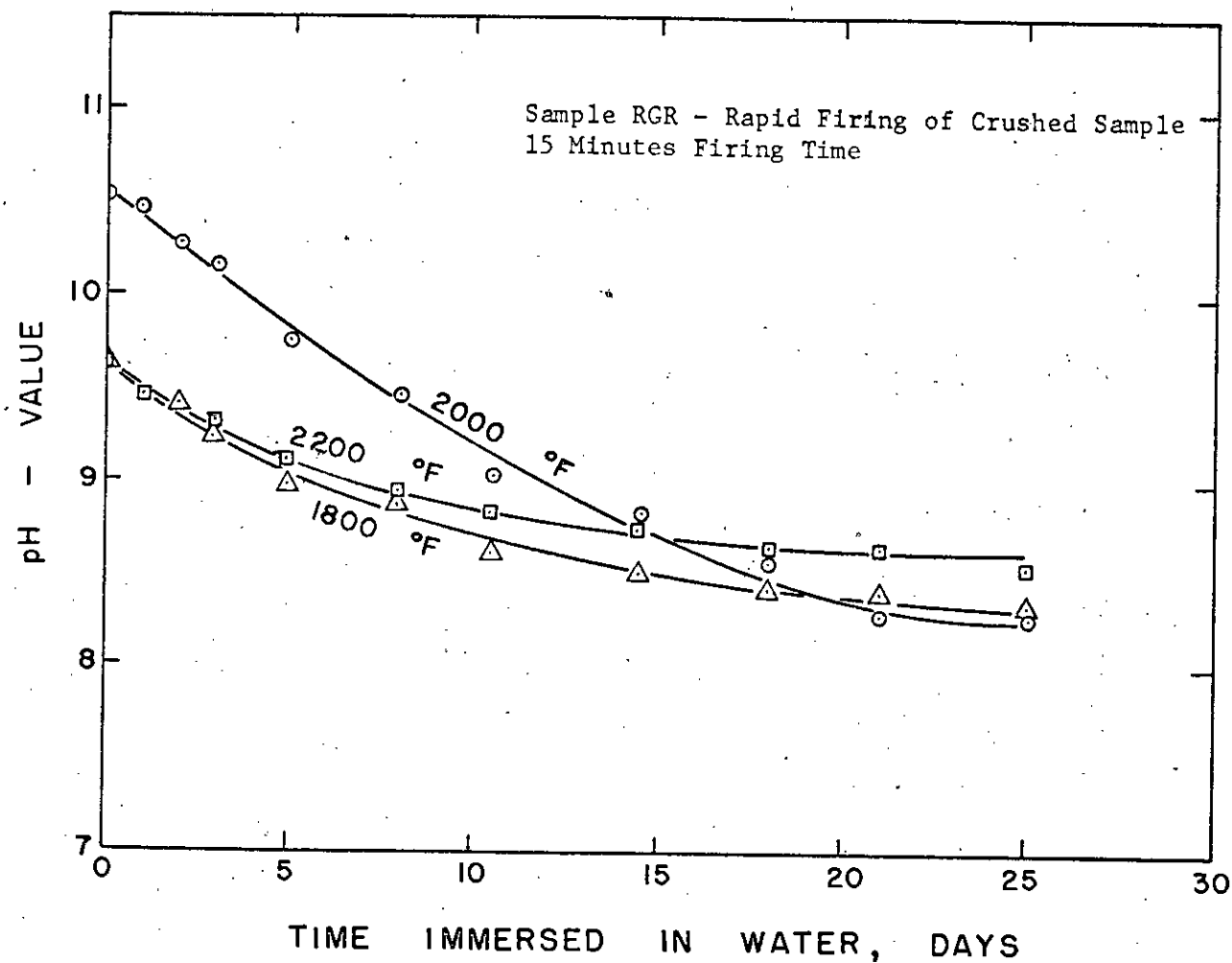


Fig. 39. Change in Alkalinity with Time for Sample RGR Immersed in Deionized Water.

aggregate caused appreciable expansion by alkali-aggregate reaction in non-alkali cement. The formation of such silicate glasses can be expected in most synthetic aggregates because of the presence of both potassium and sodium as salts and adsorbed cations in many clays and shales.

In recent years, concrete distress attributable to alkali-carbonate reaction has been reported from different parts of the world. This type of reaction is common in dolomitic aggregates. As dolomite was noticed in the X-ray diffractographs of samples CNV and SCT, this type of reaction might be expected in these samples fired at temperatures where complete dissociation of this mineral would not take place. The cause of expansion in concrete using this type of aggregate has been attributed to the increase in volume of dolomite in the process of dedolomitization, swelling of the colloidal materials formed by the reaction and/or the osmotic pressure built up at individual dolomite crystals by the differences in flow tendencies, through the colloidal material, of the alkali-carbonate solution produced in the dedolomitization reaction and pores solutions containing the alkali hydroxide (35).

From the preceding discussion, it is evident that a complete dissociation of all impurities which are essential for bloating clays and shales would improve the quality of the fired products. It is expected that such complete dissociation could be achieved by using slow firing rather than rapid firing.

Rapid firing would result in non-uniform burning of the material and incomplete dissociation of some minerals at firing temperatures much higher than temperatures at which those minerals would dissociate under slow firing. Exothermic peaks at about 900°F in DTA patterns (Fig. 40) for sample DLS fired at 1800 and 2000°F under rapid firing for 15 minutes indicate the presence of such impurities. Slow firing might cause a reduction in bloating. However, for achieving an inert material, such firing appears to be a must for some materials containing a high amount of impurities.

It may also be remarked here that before accepting synthetic aggregates as a construction material for concrete structures, a detailed study should be made to determine whether the material will cause an excessive expansion and any other deleterious reaction with cement.

6.2.6. Compressive Strength and Modulus of Elasticity.

Table IX represents the results of compression tests for samples RGR and DLS fired at different temperatures. Each sample showed an optimum firing temperature at which both compressive strength and modulus of elasticity were maximum. This temperature, as shown in Figs. 41 and 42, was about 1850°F for sample RGR and about 1800°F for DLS. Above this temperature each sample showed a decrease in both compressive strength and modulus of elasticity with an increase in porosity (Fig. 43). The rate of this decrease in compressive strength and modulus of elasticity was

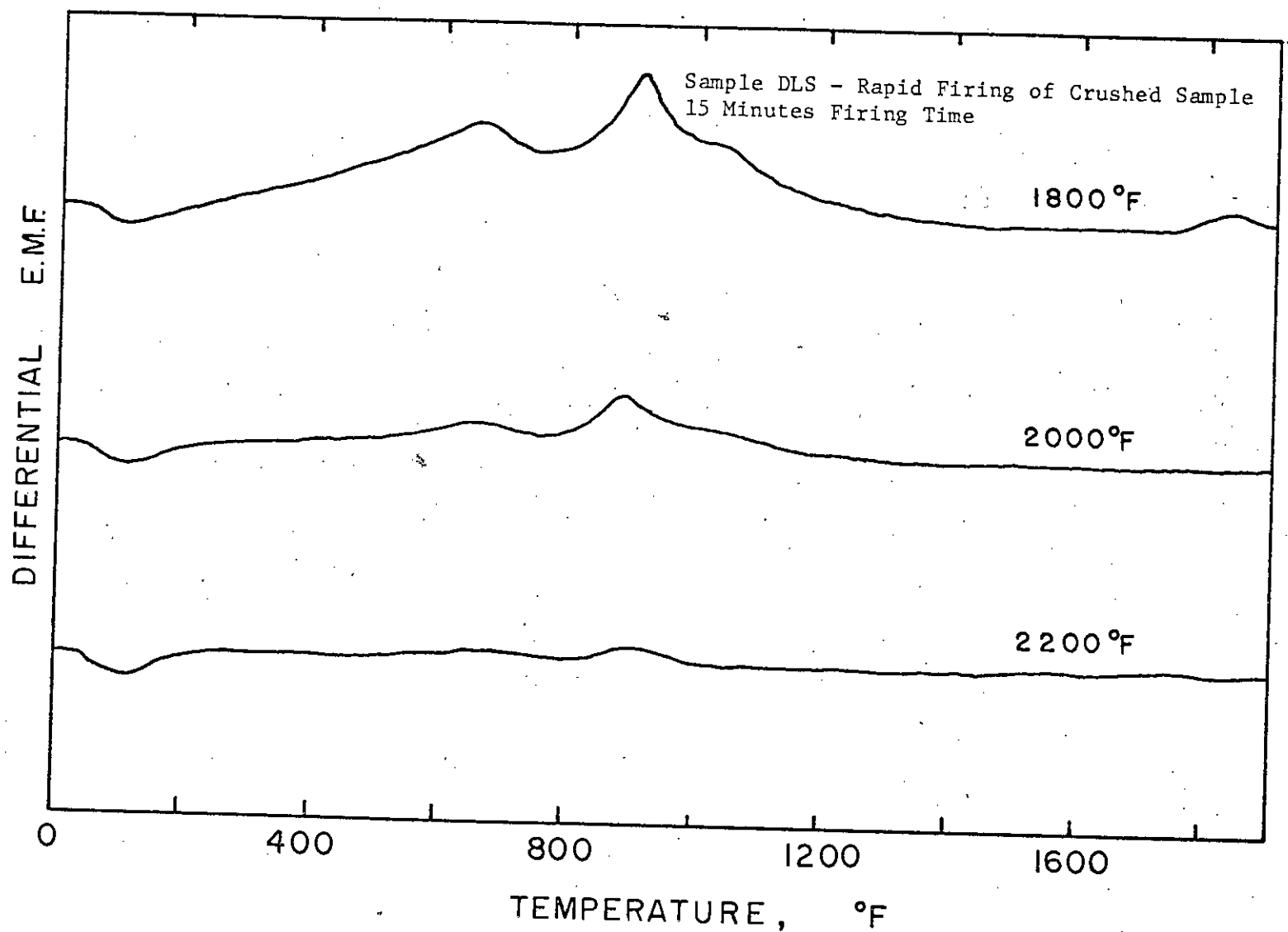


Fig. 40. Differential Thermal Curves for Sample DLS Fired at Different Temperatures.

Table IX. Compressive Strength and Modulus of Elasticity of Fired Samples.

Sample	Firing Temper- ature F°	Compressive Strength			Tangent Modulus of Elasticity, ksi					
		ksi			At 25% Ultimate Strength			At 50% Ultimate Strength		
		Average	Maximum	Minimum	Average *	Maximum	Minimum	Average *	Maximum	Minimum
RGR	1600	5.19	5.47	4.85						
	1750	6.26	6.99	5.80	710	760	660	1130	1200	1070
	1800	8.18	9.32	7.65	710	720	690	1020	1070	970
	1850	8.85	9.56	8.37	920	960	870	1320	1370	1250
	1900	6.40	6.86	5.91	980	1170	880	1340	1440	1140
	1950	5.06	5.30	4.73	820	840	800	1080	1120	1060
	2000	4.04	4.40	3.49	730	760	680	990	1020	950
DLS					600	630	560	840	870	800
	1600	5.29	5.82	4.52	790	830	700	1260	1320	1210
	1750	5.62	6.15	4.75	830	850	800	1280	1310	1250
	1800	6.51	7.20	5.77	940	1030	830	1440	1570	1310
	1850	5.78	6.56	5.10	860	940	770	1290	1410	1180
	1900	4.76	5.19	4.06	700	730	660	1020	1080	980
	1950	3.99	4.39	3.61	620	650	600	920	960	880
	2000	3.19	3.62	2.86	520	540	500	740	770	690

*Average of seven specimens.

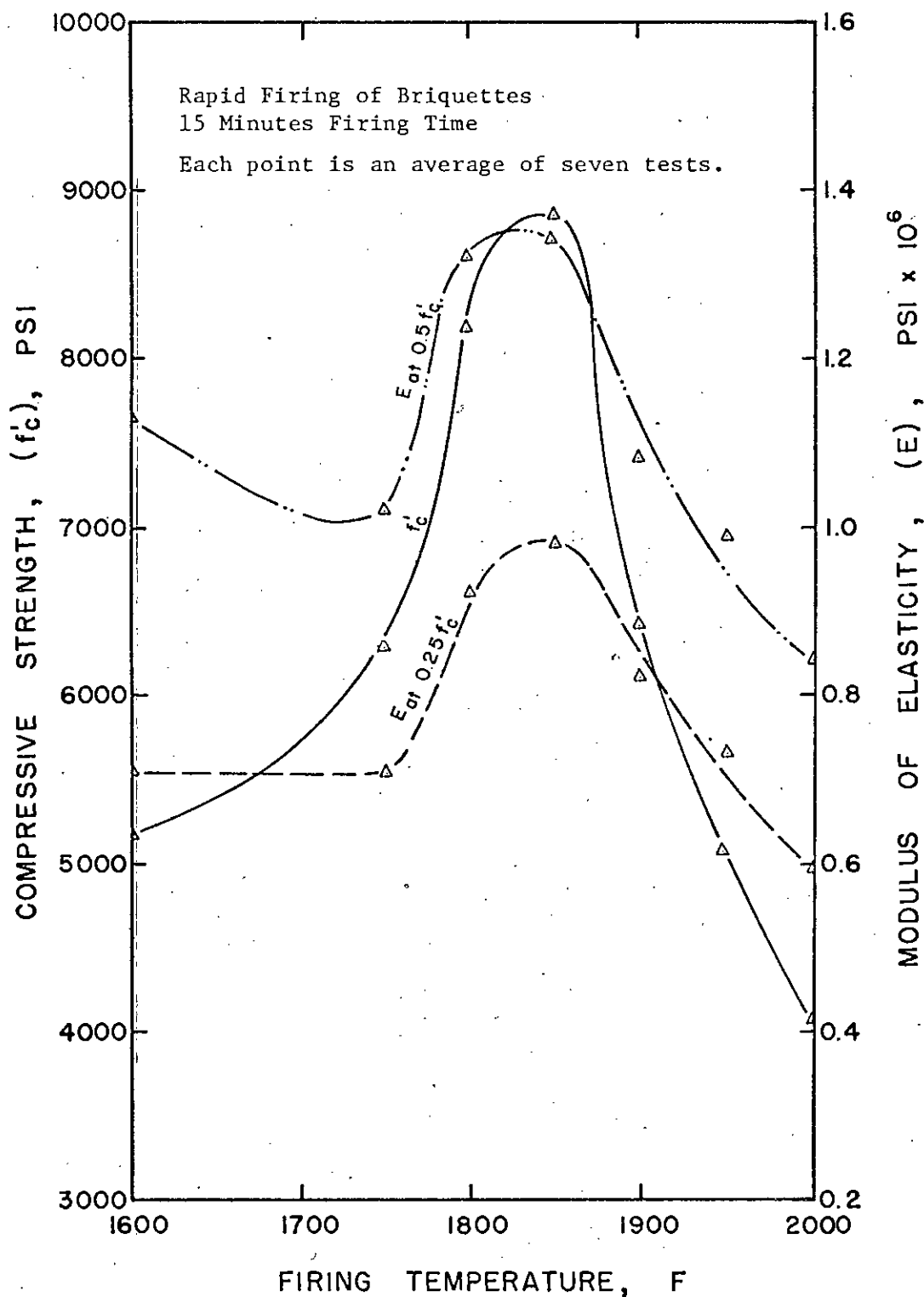


Fig. 41. Compressive Strength -- and Modulus of Elasticity -- Firing Temperature Curves for Sample RGR.

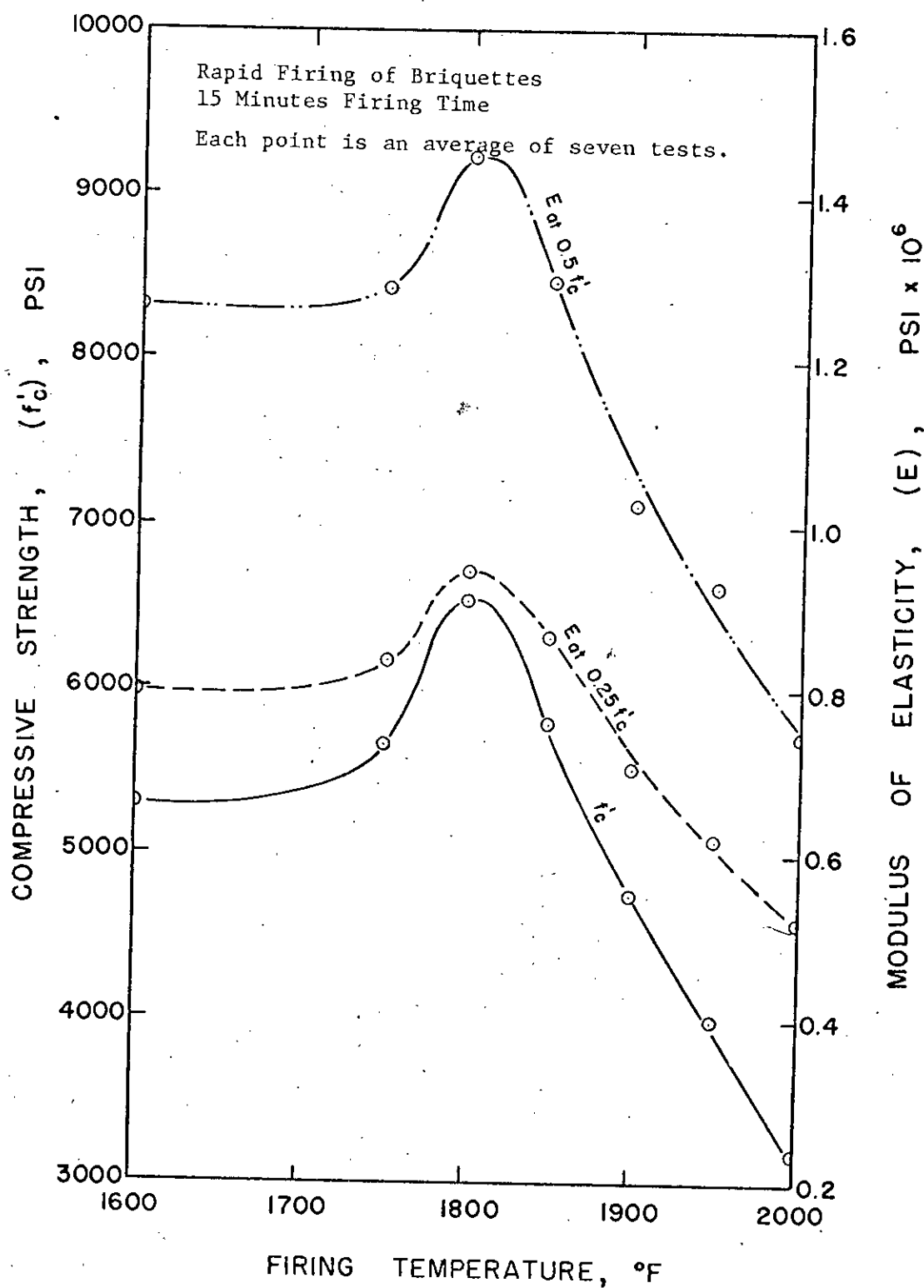


Fig. 42. Compressive Strength -- and Modulus of Elasticity -- Firing Temperature Curves for Sample DLS.

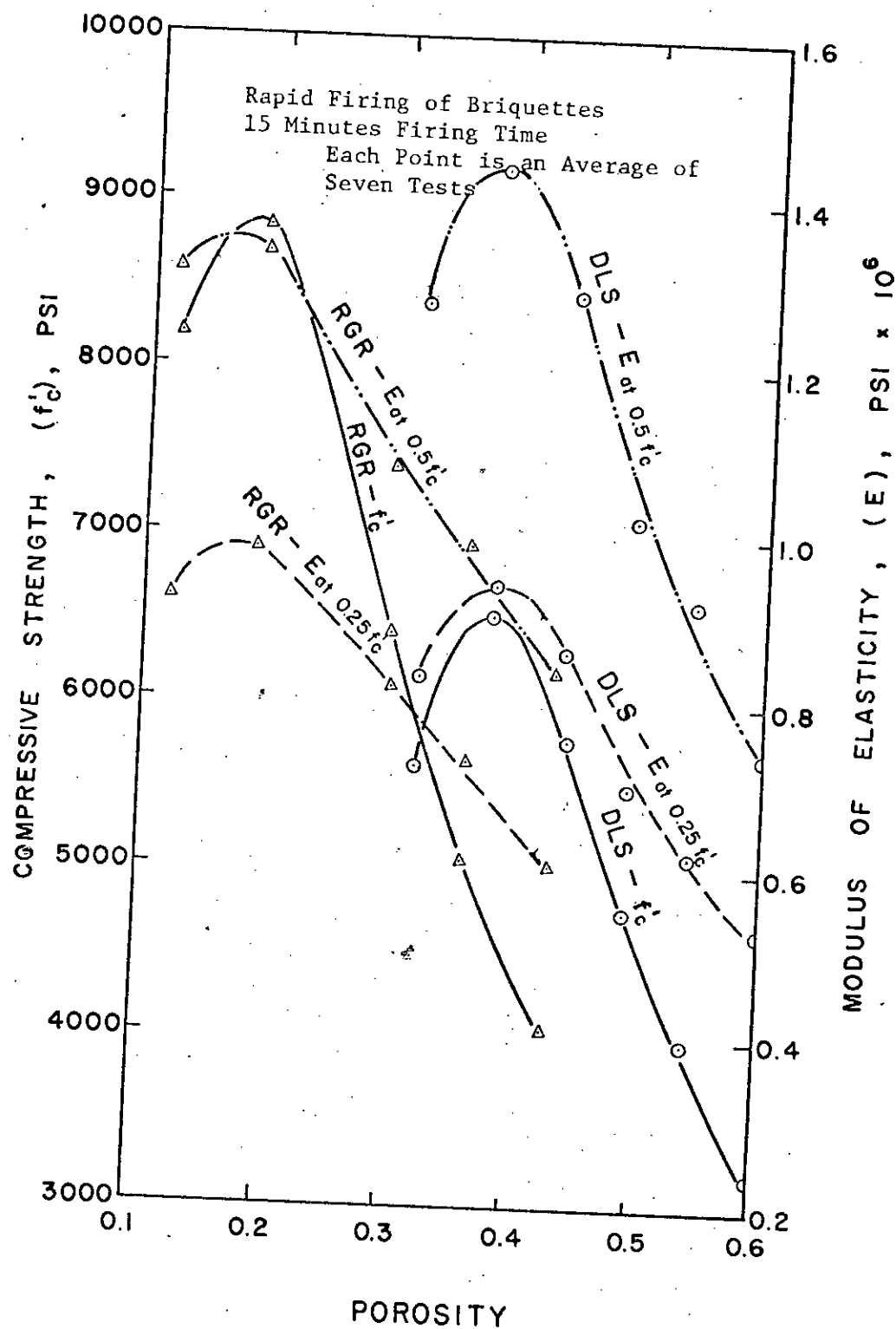


Fig. 43. Variation of Compressive Strength and Modulus of Elasticity with Porosity for Samples RGR and DLS.

higher than the rate of increase in porosity with firing temperature. This rapid decrease in these values with a slow increase in porosity was probably because of increased stress concentrations at higher porosities causing an uneven stress distribution across the cross-section

The sharp increase in both compressive strength and modulus of elasticity at about 1800°F for sample DLS and about 1800°F for sample RGR was accompanied by the development of a new high temperature phase within each material resulting in a product with glassy or vitrified bonds. This high temperature reaction was also noticed in the DTA pattern of each sample.

It is known that the strength of a material increases with a decrease in porosity and an increase in true specific gravity. To achieve higher strength of a material, it is, therefore, desirable to limit its porosity to as low as possible a value through an increase in the specific gravity. As discussed in section 6.2.1., the true specific gravity, or absolute density, of each sample increased with firing temperature and attained its maximum value at temperature above 2000°F. The increase in porosity or bulk specific gravity of sample DLS was noticed at temperature as low as 1200°F. Such increase in porosity of sample RGR took place at temperature above 1800°F. This difference in expansion characteristics, caused by the difference in their mineralogical composition, resulted a significant difference in their mechanical properties.

Figures 42 and 43 illustrate that both compressive strength

and modulus of elasticity showed similar variations with firing temperature and porosity. No correlation was noticed between these two properties. The rate of change of both compressive strength and modulus of elasticity with porosity for each sample showed similar variation. However, to make any conclusive remarks, more extensive work is needed. Sample DLS having a lower compressive strength showed a higher modulus value than sample RGR with higher compressive strength at any particular firing temperature. This difference in their behaviors may be caused by the differences in their microstructures and mineralogical compositions.

Figure 44 illustrates typical stress-strain (compression) curves for samples RGR fired at different temperatures. The lower slopes and non-linearity of these curves at low stress levels were probably caused by an initial viscous flow of the glassy phase in the material. The presence of microcracks formed in the process of rapid firing and cooling may also cause such a flow in the material. At higher stress levels, the stress-strain variation was more linear. However, the tangent moduli at 25 per cent ultimate strength were lower than that at 50 per cent ultimate strength, the latter being, in general, 50 per cent greater than the former.

It may be remarked here that since the preceding results were obtained by testing briquettes made from remolded materials, a difference in variations in these properties with firing

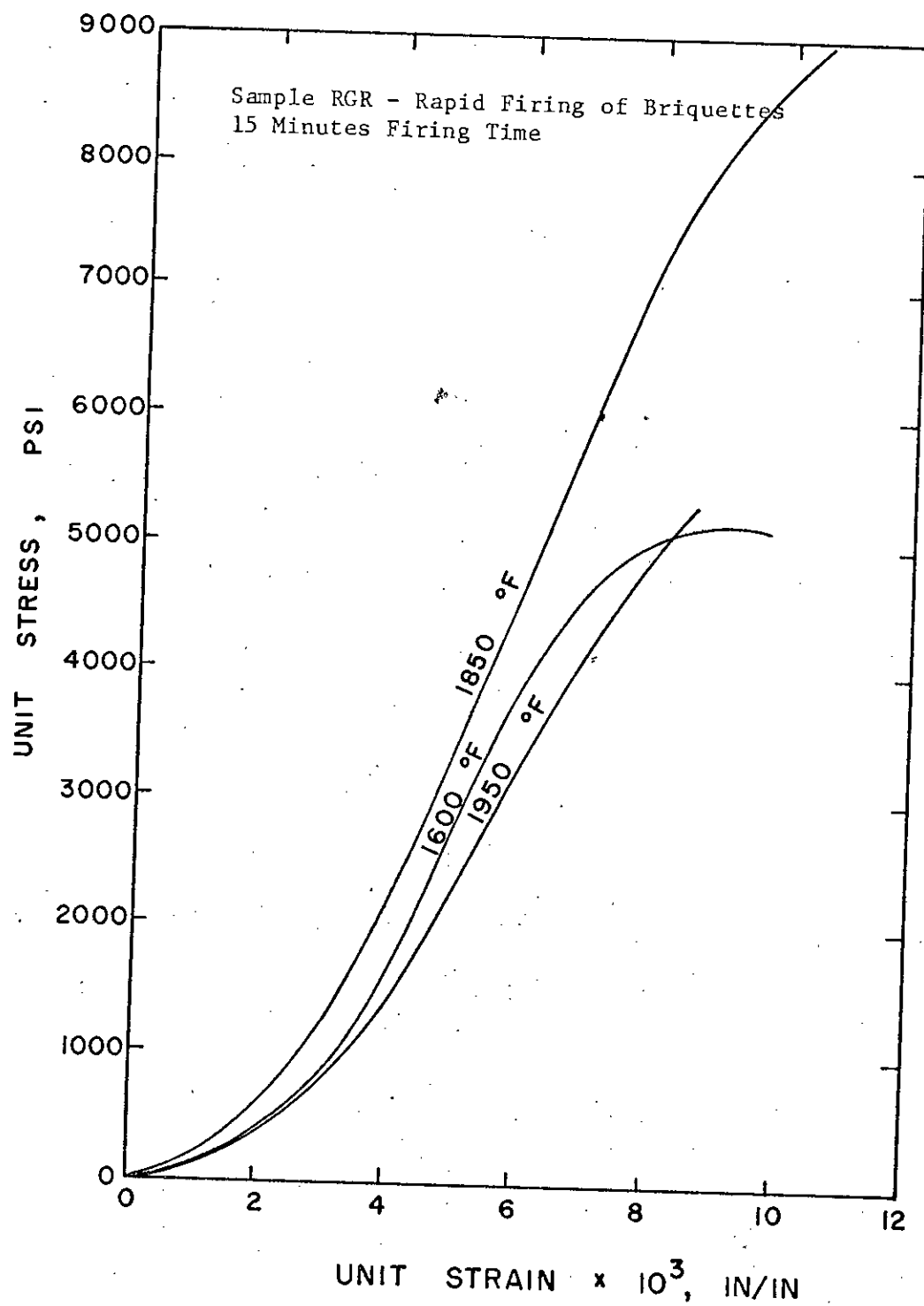


Fig. 44. Stress-Strain Curves for Sample RGR.

temperature may be expected when crushed materials are used, because of the presence of laminations and non-uniform distributions of fluxing and gas-producing minerals in the raw materials.

CHAPTER VII

SUMMARY OF CONCLUSIONS AND RECOMMENDATIONS

7.1. Conclusions

As a result of this laboratory investigation on the fundamental properties and mechanical behaviors of fired clays and shales, the following conclusions were drawn:

1. A study of the mineral contents of raw materials and their changes with firing temperature provides data which help define the fundamental properties of synthetic aggregates.
2. X-ray diffraction and differential thermal analyses supplement each other in determining mineralogical compositions of clays and shales.
3. Petrographic examinations of synthetic aggregates can be used to determine the relative amounts of crystalline and glassy phases and provide a means whereby the microstructure of these materials can easily be observed.
4. Differential thermal analyses of untreated samples provide the dissociation temperatures of different impurities in the raw material. Such information is necessary to predetermine the approximate temperature range to be used in actual firing of the material in the kiln.

5. Differential thermal analyses of fired samples indicated the presence of some non-dissociated minerals in the synthetic products fired under rapid firing at temperatures much higher than the slow fired dissociation temperatures of those minerals. Such minerals may cause poor durability of the products and also make them reactive. The presence of these minerals calls for a slow firing for such materials.
6. The transformations that occur in mineralogical compositions of a material in the process of firing are greatly influenced by the impurities and rate of heating.
7. Different bloaters having nearly the same clay mineral compositions showed similar trends in changes of their bulk specific gravity and absorption with firing temperature.
8. Clays and shales that bloated under rapid firing also bloated under slow firing. The difference in bloating under the two processes was different for different samples. For some samples, slow firing caused higher bloating at lower temperatures than that under rapid firing.
9. Impurities, such as organic matter with or without sulfur, which dissociate at temperatures be-

tween 650 and 900°F can cause bloating of clays and shales at about 2000°F under rapid firing if a viscous mass is formed.

10. No significant difference in bloating was noticed in increasing the firing time from 8 minutes to 15 minutes. Hence, it may be concluded that, for all samples tested, most of the expansion took place within first eight minutes of firing.
11. Observation indicated that an excess amount of free water in the raw material caused the material sample to explode in the furnace at elevated temperatures. Such explosions could be avoided by pre-drying the raw material at a pre-determined temperature.
12. Remolding of clays and shales improved their bloating characteristics with a reduction in water absorption. This improvement was primarily due to a uniform distribution of gas-producing minerals during remolding.
13. Water absorption of synthetic products fired above 1800°F continued for more than 28 days, the rate of absorption being high initially. In most samples, 40 to 50 per cent of 28-day absorption took place within the first 24 hours.
14. Absorption of any sample within a particular

period as a per cent of 28-day absorption did not change appreciably with firing temperature above 1900°F, although the 28-day absorption of samples fired at 2200°F was about four to five times higher than the 28-day absorption of the same sample fired at 1900°F.

15. The resistance to freeze-thaw of aggregate particles was increased with an increase in firing temperature from 1800 to 2200°F. This increase in resistance with firing temperature was primarily caused by the formation of a vitrified mass at higher temperatures. Failure of aggregate particles fired at lower temperatures was caused by splitting of particles along their planes of lamination. In general, smaller particles were more resistant than larger particles.
16. The modified Los Angeles abrasion test of fired samples indicated the presence of an optimum temperature for each sample at which the per cent wear was a minimum. This temperature for both samples tested was about 2000°F. Calculations based on the fractions passing through No. 4 and No. 12 sieve should be used for such materials. The difference between these two fractions is a measure of brittleness of the

material. The fracture of aggregate particles fired at low temperatures took place along their planes of lamination. At higher temperatures, each sample was noticed to be more brittle than at lower temperatures.

17. The fracture of aggregate particles along their planes of lamination calls for a higher firing temperature of the material to form a more completely vitrified mass. Under laboratory firing conditions, such temperature appears to be about 2000°F for the shales tested.
18. In the process of cooling care must be taken to avoid sharp temperature gradients between the surface and inside of a particle. Such temperature gradients result in excessive cracks along the surface of each particle.
19. The ASTM test method for potential reactivity of aggregate (ASTM Designation C289-64) indicated the non-reactivity of each sample fired at temperatures between 1800 and 2200°F except sample RGR fired at 2200°F, which was indicated as reactive. However, this test is not conclusive.
20. Because of the high alkalinity of synthetic aggregates, alkali aggregate reaction may be expected, even with non-alkali cement. Fourteen-day soaking

of synthetic products of each sample in water resulted in a reduction in pH value from about ten to eight, indicating a great reduction in alkalinity. Such soaking may also reduce the severity of alkali aggregate reaction, if any.

21. A complete dissociation of most of the impurities in a material should be achieved to increase the durability of synthetic products. A 15-minute firing time was found to be insufficient to dissociate some impurities (dissociation temperature about 800°F) even at 2000°F under laboratory conditions. Dissociation of such impurities appears to depend primarily on firing atmosphere.
22. Both compressive strength and modulus of elasticity of each remolded sample attained maximum values at an optimum temperature which closely corresponded to the first high temperature exothermic peak in the DTA pattern of the sample.

7.2. RECOMMENDATIONS

1. Differential thermal analyses should be made to temperatures about 2400°F to study the high-temperature reactions in clays and shales in the process of production of synthetic aggregates.
2. In order to make any definite conclusion regarding the potential reactivity of synthetic aggregates, mortar bar tests method (ASTM Designation C227-64) is recommended.
3. To make any conclusive remark on variation of both compressive strength and modulus of elasticity with porosity of synthetic aggregates, more extensive research is needed.
4. Lastly, it may be recommended that such laboratory test results should be correlated with the field performance of synthetic aggregates produced in the rotary kiln, if an extensive investigation is desired.

REFERENCES

1. Conley, J.E., Wilson, H., and Klienefelter, T.A., "Production of Lightweight Concrete Aggregates from Clays, Shales, Slates, and Other Materials," U.S. Bureau of Mines, R.I. No. 4401, 1948, pp. 114.
2. Riley, C.M., "Relation of Chemical Properties to the Bloating of Clays," Am. Ceramic Soc. Jour., Vol. 34, 1951, pp. 121-128.
3. Murray, H.H., and Smith, J.M., "Lightweight Aggregate Potentials of Some Indiana Shales," Geological Survey, Indiana Department of Conservation, Report of Progress No. 12, 1958, pp. 40.
4. Grim, R.E., Applied Clay Mineralogy, McCraw-Hill Book Co., Inc., New York, 1962, pp. 37-43.
5. Jackson, T.E., Discussion of the paper "Changes in Color of Clays on Ignition in Clay Ware Kilns," by Arthur Hopwood, Ceramic Soc. Trans., 1903, pp. 37-43.
6. Orton, E., Jr., and Staley, H.F., "Status of Carbon, Iron, and Sulfur in Clays During Various Stages of Burning," Natl. Brick Manufacturers Assoc., 3d Rept., Indianapolis, T.H. Randall and Co., 1908.
7. Wilson, H., Ceramics - Clay Technology, McGraw-Hill Book Co., New York, 1927, pp. 296.
8. Conley, J.E., Wilson, H., and Klienefelter, T.A., "Production of Lightweight Concrete Aggregates from Clays, Shales, Slates and Other Materials," U.S. Bureau of Mines, R.I. No. 4401, 1948, pp. 57-62.
9. Riley, C.M., "Relation of Chemical Properties to the Bloating of Clays," Am. Ceramic Soc. Jour., Vol. 34, 1951, pp. 121-128.
10. Lawrence, W.F., Jr., A Study of the Effect of Various Factors on the Bloating of Some Naturally Occurring and Synthesized Materials, M.S. Thesis, The New York State College of Ceramics, June, 1949.
11. Conley, J.E., Wilson, H., and Klienefelter, T.A., "Production of Lightweight Concrete Aggregates from Clays, Shales, Slates, and Other Materials," U.S. Bureau of Mines, R.I. No. 4401, 1948, pp. 21.

12. Edelman, C.H., and Favejee, J.C.L., "On the Crystal Structure of Montmorillonite and Halloysite," Z. Krist., Vol. 102, 1940, pp. 417-431.
13. Taylor, H.W.F., "Crystallographic Aspects of High Temperature Transformations of Clay Minerals," Proc. 12th Natl. Conf. on Clays and Clay Minerals, The Macmillan Co., New York, 1964, pp. 9-10.
14. Wahl, F.M., and Grim, R.E., "High Temperature D.T.A. and X-ray Diffraction Studies of Reactions," Proc. 12th Natl. Conf. on Clays and Clay Minerals, The Macmillan Co., New York, 1964, pp. 69-81.
15. Ibid, pp. 73-77.
16. Grim, R.E., Applied Clay Mineralogy, McGraw-Hill Book Co., Inc., New York, 1962, pp. 94.
17. Wahl, F.M., and Grim, R.E., "High Temperature D.T.A. and X-ray Diffraction Studies of Reactions," Proc. 12th Natl. Conf. on Clays and Clay Minerals, The Macmillan Co., New York, 1964, pp. 73.
18. Grim, R.E., Applied Clay Mineralogy, McGraw-Hill Book Co., Inc., New York, 1962, pp. 87.
19. Grim, R.E., and Kulbicki, G., "Montmorillonite: High Temperature Reactions and Classification," Am. Mineralogist, Vol. 46, 1961, pp. 1329-1369.
20. Grim, R.E., and Bradley, W.F., "Effect of Heat on Illite and Montmorillonite," Am. Ceramic Soc. Jour. Vol. 23, 1940, pp. 242-248.
21. Wahl, F.M., "Effect of Impurities on Kaolinite Transformations as Examined by High-Temperature X-ray Diffraction," Advances in X-ray Analysis, 10th Conf., Plenum Press, New York, 1962, pp. 264-274.
22. Cottrell, A.H., The Mechanical Properties of Matter, John Wiley & Sons, Inc., New York, 1963, pp. 86.
23. Hashin, Z., "The Elastic Moduli of Heterogeneous Material," Jour. of Applied Mechanics, Vol. 29, 1962, pp. 143-150.

24. Van Vlack, L.H., Physical Ceramics for Engineers, Addison-Wesley Publishing Co., Inc., Massachusetts, 1964, pp. 133.
25. Cottrell, A.H., The Mechanical Properties of Matter, John Wiley & Sons, Inc., New York, 1963, pp. 209.
26. Dieter, G.E., Jr., Mechanical Metallurgy, McGraw-Hill Book Co., Inc., New York, 1961, pp. 194.
27. Coble, R.L., and Kingery, W.D., "Effect of Porosity on Physical Properties of Sintered Alumina," Am. Ceramic Soc. Jour., Vol. 39, 1956, pp. 377.
28. Freas, D.H., "Occurrence, Mineralogy, and Origin of Lower Golden Valley Kaolinitic Clay Deposits Near Dickinson, North Dakota," The Geol. Soc. of Am., Bull., Vol. 73, No. , 1962, pp. 1343-1345.
29. Rushing, H.B., "Lightweight Aggregate Abrasion Study," Research Project 61-7C Report, Bureau of Public Roads, Louisiana Department of Highways, Feb., 1963.
30. Manual of Testing Procedures, of the Texas Highway Department, Vol. 1 and 2. Revised February, 1963.
31. Meininger, R.C., Fox, J.F., and Lepper, H.A., Jr., "A Single-Particle Freezing Resistance Test for Concrete Aggregate," ASTM, Proc., Vol. 65, 1965, pp. 801-808.
32. Gallaway, B.M., "Interim Report on the Use of Expanded Shale and Precoated Limestone as Coverstone for Seal Coats and Surface Treatments," Research Report No. 51-1, Texas Transportation Institute, Texas A&M University, 1964, pp. 51-60.
33. Mielenz, R.C., Greene, K.T., and Benton, E.J., "Chemical Test for Reactivity of Aggregates with Cement Alkalies: Chemical Processes in Cement-Aggregate Reaction," Proc., Am. Concrete Inst., Vol. 44, 1948, pp. 193.
34. Pike, R.G., Hubbard, D., and Newman, E.S., "Binary Silicate Glasses in the Study of Alkali-Aggregate Reaction," Highway Research Board, Bull. 275, 1960, pp. 39-44.
35. Hadley, D.W., "Alkali Reactivity of Dolomite Carbonate Rocks," Highway Research Board, Record No. 45, 1964, pp. 1-20.

A P P E N D I X A

EXCERPT FROM
THE DETERMINATION OF THE MOISTURE ABSORPTION
CHARACTERISTICS OF LIGHTWEIGHT CONCRETE
AGGREGATES

A Thesis

By

JULIAN STEPHEN BRYANT

TEXAS A&M UNIVERSITY

JANUARY, 1959

EXCERPT FROM
THE DETERMINATION OF THE MOISTURE ABSORPTION
CHARACTERISTICS OF LIGHTWEIGHT CONCRETE
AGGREGATES

PART III

A New Method Of Test

The purpose of this research was to devise a simple, reliable method of test that would determine values of absorption, rate of absorption, and specific gravities for both fine and coarse fractions of lightweight aggregates. This objective has been accomplished in the method outlined below.

The Theory

It was necessary to devise a method of test which did not require handling of the sample or obtaining a saturated, surface-dry condition physically in the sample, and also one which would determine the rate of absorption. The approach was to devise a method that would give the saturated weight of the sample and the surface-dry volume without actually obtaining this condition at the same time.

If the sample is immersed in water in a container of known volume and the water is maintained at a constant level by adding water as the sample absorbs it, then the rate of absorption can be measured by weighing the container at specific time intervals during the test. Then if a rate curve can be established and extrapolated to include zero time, the total

amount of absorption can be obtained. Also, the surface-dry volume of the sample can be obtained by subtracting the volume of water at zero time from the volume of the container.

The Test

Scope. This method of test is intended for use in determining the bulk specific gravity, both dry and saturated, surface-dry, apparent specific gravity, absorption (after 72 hours at room temperature), and rate of absorption of both the coarse and fine lightweight concrete aggregates. The specific gravity values are as defined in the Standard Definitions of Terms Relating to Specific Gravity (ASTM Designation: E 12) of the American Society for Testing Materials.

Apparatus. The apparatus shall consist of the following:

- (a) Balance. -- A balance having a capacity of 1kg. or more and a sensitivity of 0.1 gm. or less.
- (b) Container. -- A glass Mason jar fitted with a conical brass cap with a hole one-quarter inch in diameter in the top, as illustrated in Fig. 45.

Sample. Approximately 200 gm. of the aggregate shall be selected by the method of quartering from the sample to be tested.

Procedure. The procedure shall be as follows:

- (a) The jar and cap shall be weighed to the nearest 0.1 gm. The jar shall then be filled completely with distilled water and weighed to the nearest 0.1 gm. and the temperature of the water recorded.

- (b) The sample shall be dried in an oven at a temperature of 212 to 230°F for 24 hours. It shall then be allowed to cool to room temperature in a dessicator and the weight determined to the nearest 0.1 gm.
- (c) After weighing, the sample shall be placed in the Mason jar and the jar filled with distilled water. The cap shall then be placed on the jar and water added to fill the jar and cap completely. The jar with sample and water shall then be weighed to the nearest 0.1 gm. and the temperature recorded. With a little practice, this first weighing can be accomplished two minutes after the water is first introduced into the container. Weighings shall then be made at intervals of 2, 4, 6, 8, 10, 20, 30, 60, 90, and 120 minutes from the beginning of the test and each 24 hours thereafter for a period of at least 3 days, taking care to agitate the sample by rolling and shaking the jar to remove any air trapped between the particles and refilling the jar so that a constant volume is maintained before each weighing is made.

Calculations. The weight of total water in the container at any time can be obtained by subtracting the weight of the container and the oven-dry weight of the sample from the total weight of the sample, container and water at that time. The weight of total water for each of the time intervals shall be calculated. Then, if the time intervals are represented by $t_1, t_2, t_3, \dots, t_t$ and the weights of total water corresponding to those intervals are represented by $w_1, w_2, w_3, \dots, w_t$, a curve can be plotted with time as the abscissa and total water as the ordinate which appears to be a hyperbola. If the curve approximates a hyperbola, then a plot of t versus $(t_n - t_1)/(w_n - w_1)$ will approximate a straight line. The values of $t_n - t_1, w_n - w_1$ and $(t_n - t_1)/(w_n - w_1)$ shall be calculated

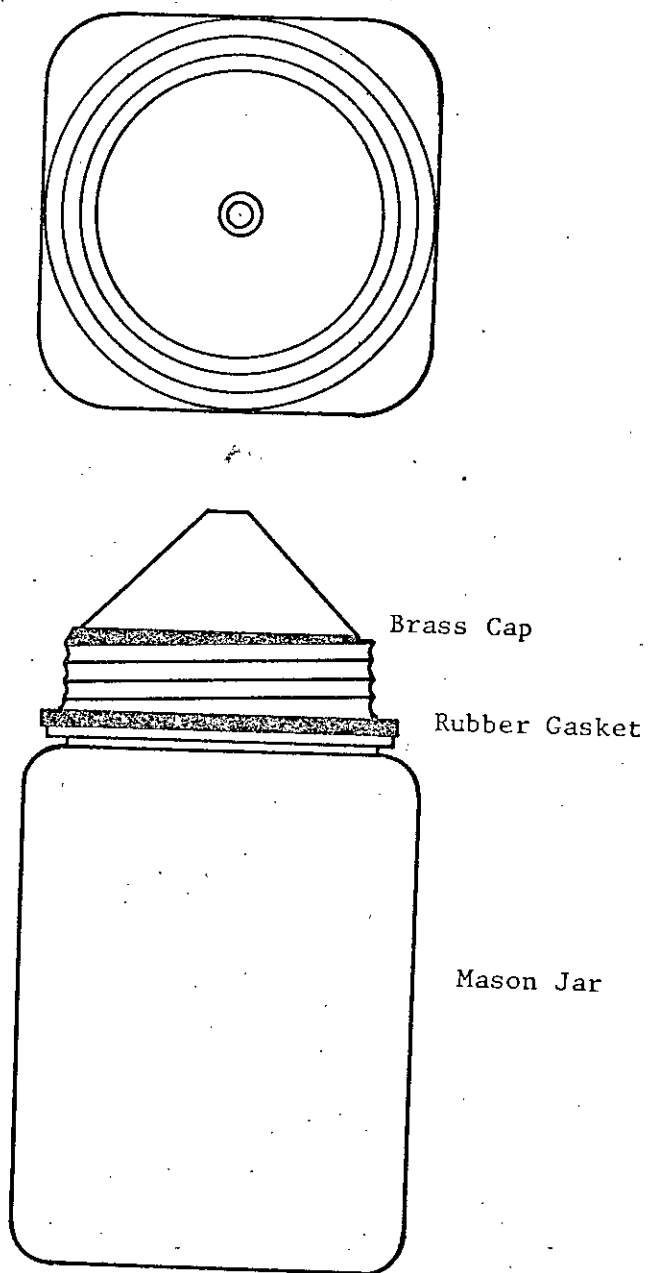


Fig. 45. Pycnometer Bottle Used in Tests.

for each time interval during the first two hours of the test and tabulated with the corresponding values of t and w . An equation of the form

$$\frac{t_n - t_1}{w_n - w_1} = a + bt_n$$

shall then be written for each value of t . These equations shall be divided in half or nearly half and the first and second halves added together separately so as to produce two remaining equations in which a and b are unknown. These two equations can be solved simultaneously and the values of a and b determined. After the values of a and b are known, the value of w_0 , that is the weight of total water at zero time, can be calculated from the relation.

$$w_0 = w_1 - \frac{t_1}{a}$$

The total water at zero time shall be referred to as the free water. For purposes of this test, free water is defined as all water in the container which is not absorbed by the sample. Assuming that the volume of the sample remains constant, then the amount of free water is constant throughout the test. In the practical application, the free water is defined as that water present in the concrete mixture which is free to combine chemically with the cement; that is, all mixing water that is not absorbed by the aggregates. The volume of free water can then be calculated by dividing the weight of free water by the specific gravity of water at the temperature recorded when the

test began. The bulk volume of the sample shall be calculated by subtracting the volume of free water from the volume of the container. The volume of total water at 3 days shall be calculated by dividing the weight of total water by the specific gravity of water at the temperature recorded at the end of the test. The apparent volume of the sample can then be calculated by subtracting the volume of total water at 3 days from the volume of the container. The absorbed water at any time can be calculated by subtracting the free water from the total water at that time.

Absorption. The percent absorption shall be calculated for each time interval by dividing the weight of absorbed water at each interval by the oven-dry weight of the sample. The percent absorption versus time shall be plotted on rectangular co-ordinate graph paper and a smooth curve drawn connecting these points to establish the rate of absorption.

Bulk Specific Gravity. The bulk specific gravity shall be calculated by dividing the oven-dry weight of the sample by the bulk volume of the sample.

Bulk Specific Gravity (Saturated, Surface-Dry). The bulk specific gravity on a saturated, surface-dry basis shall be calculated by dividing the sum of the oven-dry weight of the sample and the weight of absorbed water at 3 days by the bulk volume of the sample.

Apparent Specific Gravity. The apparent specific gravity

shall be calculated by dividing the oven-dry weight of the sample by the apparent volume.

Reproducibility of Results. Tests of separate samples of the same aggregate should check within 0.02 in the case of bulk specific gravity, within 5% of the total percentage of absorption in the case of absorption, within 5% of the saturated, surface-dry bulk specific gravity value in the case of saturated, surface dry bulk specific gravity, and within 5% of the apparent specific gravity value in the case of apparent specific gravity.

Discussion. The apparatus and procedure involved in this method are very simple. The calculations are somewhat more complicated than those required in the ASTM standard tests and may appear to be laborious at first. However, by tabulating the data and making the calculations systematically, they become quite simple and can be made rapidly by an experienced laboratory technician. The method of calculating the weight of free water is a more accurate method of extrapolating the data than merely extending a curve to intercept the zero time ordinate and obtaining the value graphically. A sample data sheet is shown in Table X as an example of how the data are recorded and organized. Fig. 46 shows values of total water versus time for one test which are assumed to describe a curve that is hyperbolic in form. Also shown in Fig. 46 is a plot of t versus $(t_n - t_1)/(w_n - w_1)$, where $t_1, t_2, t_3, \dots, t_t$ represents the time intervals at which weighings were made and

$w_1, w_2, w_3, \dots w_t$ represent total water corresponding to the time intervals, which should approximate a straight line if the curve representing the rate of increase of total water is approximately hyperbolic in form. Fig. 46 also shows the true hyperbola resulting from blotting time against values of total water computed from the relation

$$w = \frac{t - t_1}{a + bt} + w_1$$

where t represents time, and w represents total water, and a and b are the constants evaluated by the method of averages explained in the preceding text on page 128. It is possible that this curve is logarithmic or exponential in form. The rate of absorption could be inversely proportional to the amount of water absorbed, in which case the curve would be logarithmic. However, during the first two hour period the rate can be very closely approximated by a hyperbola as shown in Fig. 46 and for all practical purposes this is close enough.

Following is a set of sample calculations showing how the data recorded on the sample data sheet in Table X are analyzed and the values plotted in Fig. 46 are obtained:

The data are arranged in tabular form and the computations for $t_n - t_1$, $w_n - w_1$, and $(t_n - t_1)/(w_n - w_1)$ are made,

Table X. Data Sheet Absorption and Specific Gravity

Test No: 3 Aggregate: Stafford Fine Date: 9-6-57

(a) Tare wt. 303.4 gm. Wt. of bottle and water: 877.1 gm.
 Wt. of water: $\frac{877.1 - 303.4}{.9957} = 573.7 \text{ gm.}$
 Temp. of water: 30.0°C. Vol. of bottle: $\frac{573.7}{.9957} = 576.2 \text{ cc.}$

(b) Wt. of bottle and dry sample: 503.6 gm.
 Wt. of dry sample: $\frac{503.6 - 303.4}{.9957} = 200.2 \text{ gm.}$
 Temp. at start of test: 30.0°C. Temp. at end of test: 25.2°C.

Time since start of test	wt. tare, sample, water. gm	Wt. total water. gm	Wt. free water. gm	Wt. absorbed water. gm	Per Cent absorption. %
4 min.	971.3	467.7	466.0	1.7	0.8
8 min.	972.6	469.0	466.0	3.0	1.5
10 min.	973.6	470.0	466.0	4.0	2.0
20 min.	975.6	472.0	466.0	6.0	3.0
30 min.	976.7	473.1	466.0	7.1	3.5
60 min.	979.8	476.2	466.0	10.2	5.1
90 min.	980.9	477.3	466.0	11.3	5.6
120 min.	981.8	478.2	466.0	12.2	6.1
1 day	990.9	487.3	466.0	21.3	10.6
2 day	992.3	488.7	466.0	22.7	11.3
3 day	992.6	489.0	466.0	23.0	11.5

(c) Wt. of free water: 466.0 gm
 $\frac{466.0}{.9957} = 468.0 \text{ cc.}$

Bulk vol. of sample: $\frac{576.2 - 468.0}{.9957} = 108.2 \text{ cc.}$

Wt. of total water (3 days): 489.0 gm.

Vol. of total water: $\frac{489.0}{.9970} = 490.5 \text{ cc.}$

Apparent Vol. of sample: $\frac{576.2 - 490.5}{.9957} = 85.7 \text{ cc.}$

Saturated wt. of sample: $\frac{200.2}{.9957} + \frac{23.0}{.9957} = 223.2 \text{ gm.}$

Absorption (Per Cent): $\frac{23.0}{200.2} = 11.5\%$

Bulk Specific Gravity (Dry): $\frac{200.2}{108.2} = 1.85$

Bulk Specific Gravity (SSD): $\frac{223.2}{108.2} = 2.06$

Apparent Specific Gravity: $\frac{200.2}{85.7} = 2.34$

Point	t	$t_n - t_1$	w	$w_n - w_1$	$\frac{t_n - t_1}{w_n - w_1}$
1	4	0	467.7	0	---
2	8	4	469.0	1.3	3.076
3	10	6	470.0	2.3	2.608
4	20	16	472.0	4.3	3.720
5	30	26	473.1	5.4	4.814
6	60	56	476.2	8.5	6.588
7	90	86	477.3	9.6	8.958
8	120	116	478.2	10.5	11.047

The data are then divided into two groups, the first group containing the points 2, 3, 4, and 5 and the second group containing the points 6, 7, and 8. The value of $t_n - t_1$ for point 1 is omitted because $w_n - w_1$ is zero. This point is omitted also in the succeeding calculations since division by zero is excluded from mathematics. The values of $(t_n - t_1)/(w_n - w_1)$ and t_n are substituted in the relation $(t_n - t_1)/(w_n - w_1) = a + bt_n$ forming two sets of equations. These sets of equations are added separately to form two simultaneous equations in a and b.

$$\begin{array}{rcl}
 3.076 & = & a + 8b \\
 2.608 & = & a + 10b \\
 3.720 & = & a + 20b \\
 4.814 & = & a + 30b \\
 \hline
 14.218 & = & 4a + 68b \quad (1)
 \end{array}
 \qquad
 \begin{array}{rcl}
 6.588 & = & a + 60b \\
 8.958 & = & a + 90b \\
 11.047 & = & a + 120b \\
 \hline
 26.593 & = & 3a + 270b \quad (2)
 \end{array}$$

Equations (1) and (2) are solved simultaneously and the values of a and b are determined.

$$\begin{array}{rcl}
 \frac{14.218}{4} & = & \frac{4a}{4} + \frac{68b}{4} \quad (1) \\
 \frac{26.593}{3} & = & \frac{3a}{3} + \frac{270b}{3} \quad (2)
 \end{array}
 \qquad
 \begin{array}{rcl}
 8.864 & = & a + 90b \quad (2) \\
 3.554 & = & a + 17b \quad (1) \\
 \hline
 5.310 & = & 73b \\
 b & = & \frac{5.31}{73} = 0.072
 \end{array}$$

$$a = 3.544 - (17)(0.072)$$

$$a = 3.554 - 1.224 = 2.330$$

With the values of the constants obtained, w_0 , that is the total water at zero time or the free water, may be found by the relation,

$$w_0 = w_1 - \frac{t_1}{a}$$

which in this case is

$$w_0 = 467.7 - \frac{4}{2.330} = 466.0 \text{ gm.}$$

Values of w may be computed for any value of t from the relation,

$$w = \frac{t - t_1}{a + bt} + w_1$$

and , the residual, may be calculated by subtracting the computed value of w from the observed value. The results of these calculations are shown graphically in Fig. 46.

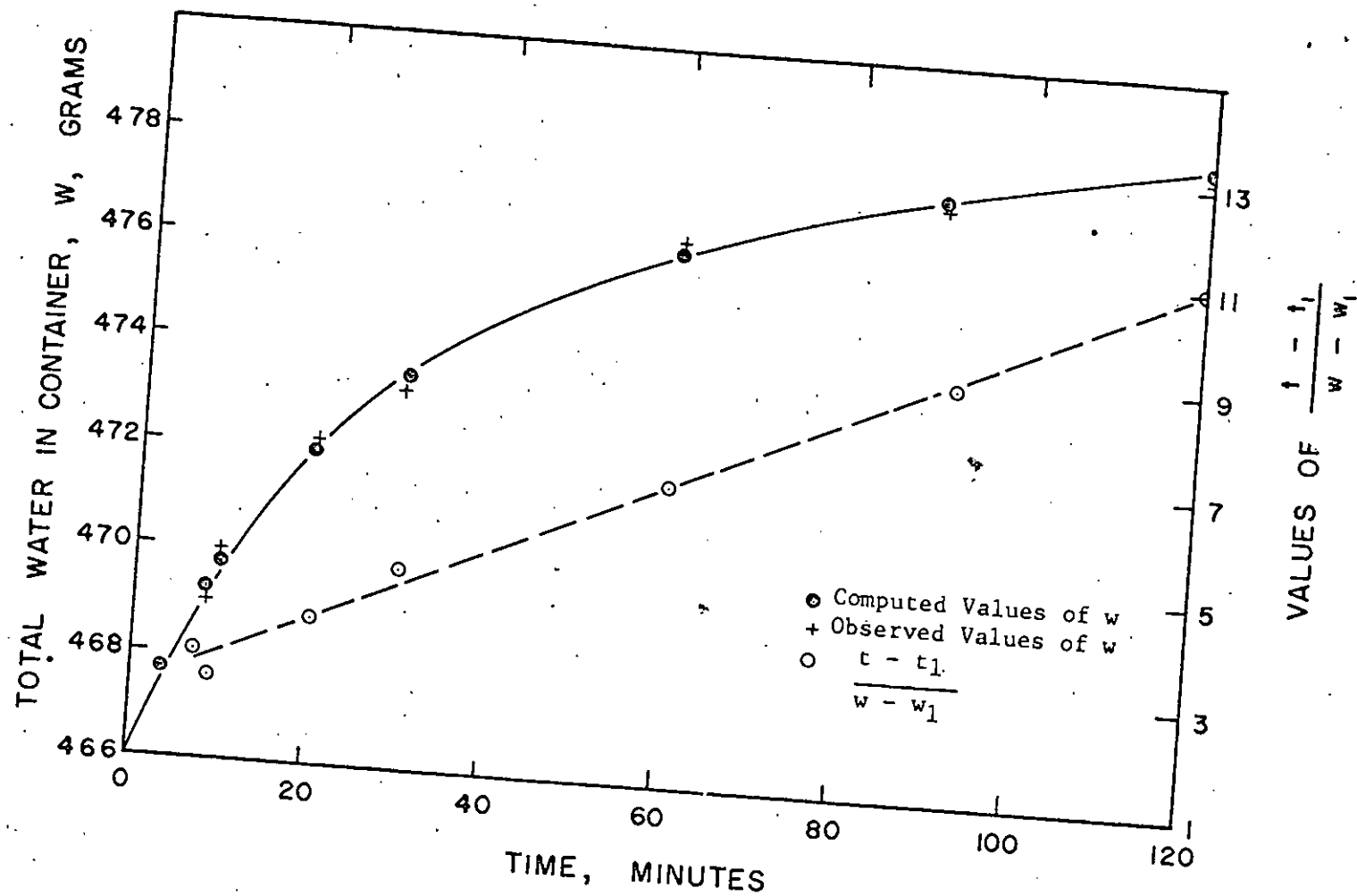


Fig. 46. Rate of Increase of Total Water During First Two Hours of Test, Showing Both Observed and Computed Values.

A P P E N D I X B

ABRASION OF CONVENTIONAL AND LIGHTWEIGHT COARSE
AGGREGATE BY THE USE OF THE
LOS ANGELES MACHINE

Test Method Tex-410-A

Revised

November 1963

ABRASION OF CONVENTIONAL AND LIGHTWEIGHT COARSE
AGGREGATE BY THE USE OF THE
LOS ANGELES MACHINE

Scope

This Test Method covers the procedure for testing conventional and lightweight coarse aggregate for resistance to abrasion in the Los Angeles testing machine with an abrasive charge. The apparatus and procedure used in this test are identical with ASTM Designation: C 131 with the exceptions noted under Part II of the method.

PART I

Abrasion Of Conventional Coarse Aggregate

Procedure

Use the apparatus specified to prepare and test the required gradings of aggregate in accordance with the procedure described in ASTM Designation: C 131.

PART II

Abrasion Of Lightweight Coarse Aggregate

Procedure

To avoid the excessive volume of material in the testing machine which will occur when the lightweight aggregate sample is prepared according to ASTM Designation C 131, it is necessary to reduce the weight proportionately to obtain an equal volume of lightweight aggregate comparable to that normally obtained

with a conventional aggregate sample.

The abrasive charge must also be reduced in a similar manner.

1. Determine the unit weight (U_L) of the lightweight aggregate by Test Method Tex-404-A.
2. Assume an average unit weight of conventional aggregate to be 97.0 lbs. per cu. ft.
3. Reduce the lightweight aggregate sample.

$$\frac{U_L}{97.0} = \frac{X}{C}$$

$$X = \frac{(C) (U_L)}{97.0}$$

Where:

U_L = Unit weight of lightweight aggregate sample
(lbs. per cu. ft.)

C = Weight of conventional aggregate required
for grading in ASTM 131

X = Reduced lightweight aggregate sample charge.

4. Reduce the abrasive charge:

$$\frac{U_L}{97.0} = \frac{X_L}{C_L}$$

$$X_L = \frac{(C_L) (U_L)}{97.0}$$

Where:

U_L = Unit weight of lightweight aggregate
(lbs. per cu. ft.)

C_L = Weight of abrasive charge required for
grading in ASTM 131

X_1 = Reduced abrasive charge for lightweight aggregate.

5. Remainder of procedure as set forth in ASTM 131.

NOTE:

It is sometimes impossible to obtain the exact abrasive charge with the steel balls available. In this case, obtain the closest abrasive charge possible to the reduced value and then adjust the weight of the sample in proportion to the new abrasive charge.

Reporting Test Results

Report the test data and type grading and the wear to the nearest 0.1 percent.

