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THESIS

FIRE CLAYS AND REFRACTORY MATERIALS

PACK

1877

MISSOURI SCHOOL
OF
MINES and METALLURGY.

FIRE CLAYS
AND
REFRACTORY MATERIALS:

A THESIS

FOR THE DEGREE
of
M.E.
By
JAS. A. PACK

Class '77.

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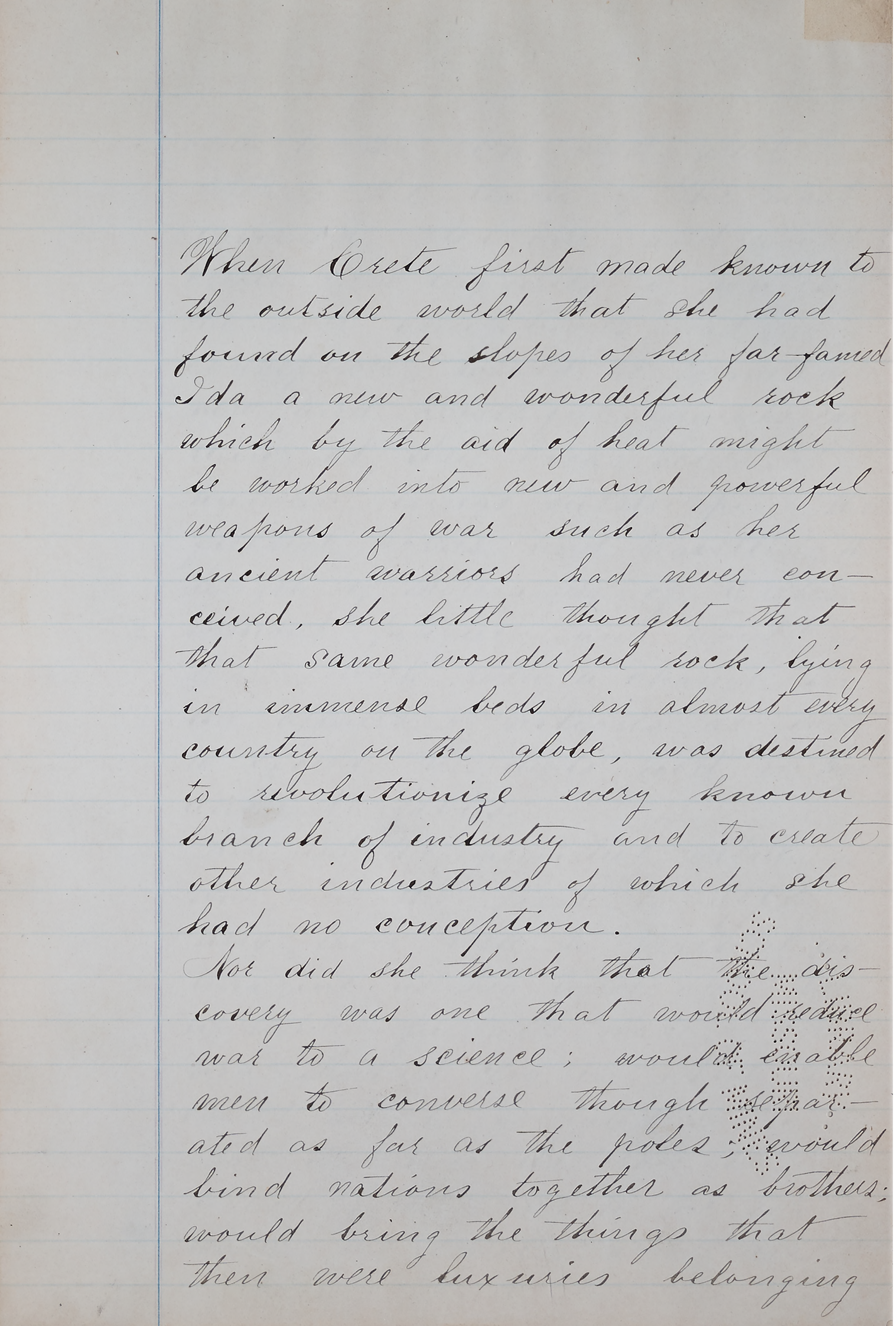

When Crete first made known to the outside world that she had found on the slopes of her far-famed Ida a new and wonderful rock which by the aid of heat might be worked into new and powerful weapons of war such as her ancient warriors had never conceived, she little thought that that same wonderful rock, lying in immense beds in almost every country on the globe, was destined to revolutionize every known branch of industry and to create other industries of which she had no conception.

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only to the rich and great within the reach of the most common plebeian, and would create new luxuries of which the most voluptuous had no conception; would be formed into new and powerful engines carrying at their backs scores of human beings, outstripping their fleetest horses and travelling the length of thousands of Cretes without fatigue; and would, in short, work such a change in the affairs of nations as even to exceed the deeds told in their ancient fables.

Nor did the primitive metallurgists who were the first to apply this iron rock to practical use, imagine, as they toiled over their unsightly furnaces made of common sundried clay in which there could not be generated sufficient heat to reduce the iron to a liquid form, and to the top of which the smelter might reach with his hand, that in the

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future this metal would need to be smelted in furnaces that would reach to the tops of their very temples, and at a temperature that would defy the resistance of the firmest granite.

But yet these things are all realized by the metallurgist of today and in his construction of the furnaces and various metallurgical apparatuses which are the outgrowth of the various discoveries of metals, and the constant invention of new methods of metallurgical treatment, there is no problem that more keenly taxes his ability than the discovery and application of the most efficient refractory materials which shall be able to endure all the varied requirements of modern metallurgy.

Indeed if we examine this subject but a moment we see that it cannot well be otherwise, for what with the immense increase of temperature incident to the introduction of many new processes (of which the Bessemer process

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for the production of steel may be cited as a single example) and with the unfitness of natural substances, and even the entire range of artificial materials to a certain degree, for the withstanding of such heat, we see nothing but continual embarassment meeting the furnace builder and retort maker whichever way he may turn.

Indeed the problem has attained such a degree of complexity through the increasing demands upon our refractory material that certain branches of industry are threatened with extinction.

The first material to which our attention is called is such as already exists in nature in the form of rocks. These rocks are mainly quartzites, granite, sandstone, serpentine, steatite, aluminous shale and sometimes carbonate of lime. The first of these has such a limited use in metallurgy that it can scarcely be admitted to

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the list of good natural refractory material. The greatest objections to it are; first, its extreme liability to crack; and, second, sensitiveness to the corrosive action of basic slags and fluxes.

Granites may also be used for furnace linings in a variety of processes provided the amount of alkalies is not excessive.

Sandstones exhibit a high degree of refractoriness, but are open to the objections of a non-homogeneous structure, and liability to corrosion by basic slags.

Serpentine and steatite may be classed among the best of natural refractory stones. They should contain 30% to 35% of magnesia and alumina.

Carbonate of lime has been used for furnace lining and may be used where the process is a continuous one but not otherwise as the lime absorbs moisture and falls to pieces

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Carbonate of lime has been used for furnace lining and may be used where the process is a continuous one but not otherwise as the lime absorbs moisture and falls to pieces

when exposed to the atmosphere.

When building the stone into the furnace wall cement of a similar nature to that of the stone should be used in order to prevent any chemical union which would cause the lines of junction of the blocks of stone to become more fusible, as in the case of siliceous rocks and lime cement.

In selecting material for furnace linings much danger to the structure might be avoided if previous to the adoption of any stone it were submitted to a thorough test as follows;

heat for several days at a low temperature a weighed piece of the stone and at the end of this time gradually raise the temperature to the highest point and maintain it thus for several hours. When the substance is removed from the fire it should not show a vitreous fracture and should have lost but little

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From what has been said, we see that nature by not furnishing the proper fire-proof materials has laid insuperable barriers in in the way of many of the most vital industries and these must be overcome by art or not at all. It is only on artificially made materials that the furnaceman of the future must depend for his linings, bottoms, retorts &c.

The manufacture of artificial fire proof material depends largely on the quality and accessability of fireclay. Of these clays there is an almost endless variety, differing in the proportions of the various constituents, and on the ratio of these constituents depends the good or bad quality of the fire clay.

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and the alkalies, but the total amount of these impurities should never exceed 5 % when a highly refractory retort or brick is desired.

The selection of the clay is of the greatest importance and should be made with great care and by means of numerous tests. Percy recommends as a test of infusibility that the clay be formed into sharp edged prisms and placed in a retort in a furnace and the temperature raised to the highest pitch. If when removed the prisms retain their sharp edges they have a good degree of refractoriness, otherwise they are inferior.

In the selection of every clay chemical analysis is also of prime importance. Although there are certain affinities or non-affinities possessed by certain clays which a chemical analysis will fail to show, yet these analyses point with a greater

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In my investigations in this field I have selected a number of specimens of Missouri clays, also of several bricks. The samples chosen are mostly from the southern part of the State.

In my analyses I have adopted the following plan as well suited to the clays under consideration, though in regard to the method of alkali determination my plan of estimation would not be suited to those clays in which the alkalies occur as chemical combinations undecomposable by acids.

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I. Estimation of water – This is determined in the absence of such constituents as carbonic acid by direct ignition, the

per cent of water being calculated directly from the loss in weight.

II. Volumetric determination of iron decomposable by sulphuric acid — A weighed amount is placed in a beaker with sulphuric acid and water and boiled 1 to 2 hours, then allowed to cool and the protoxide of iron determined by means of a standard solution of permanganate of potash. The iron is then reduced by zinc and the total iron determined. The difference between this last and the first determination gives after proper deductions for oxygen the amount of decomposable peroxide of iron in the solution.

III. Determination of remaining constituents — A weighed amount is taken and treated with sulphuric acid as before. This extracts all the decomposable salts of iron, alumina lime, magnesia and the alkalies and also carries into solution some of the soluble silica. The solution is now filtered and

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the undecomposable matter remaining on the filter is put aside for treatment. From the filtrate iron and alumina are precipitated by ammonia carrying down with them the soluble silica. This precipitate is collected on a filter, redissolved in acid, evaporated to dryness and the silica thus separated from the iron and alumina. The iron and alumina is burned and weighed and after deducting the iron found in II we have the alumina. From the filtrate remaining from the precipitation of the iron, alumina, and silica the lime is precipitated by oxalate of ammonia, collected on a filter after standing twelve hours, dried, burned, converted into sulphate by means of sulphuric acid and weighed. The remaining filtrate from lime is evaporated to dryness, all ammonia salt driven off by heat, residue dissolved in water and a few drops of acid, and caustic baryta added

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to faint alkaline reaction. This precipitates magnesia. The precipitate is collected on filter redissolved and the excess of baryta precipitated by sulphuric acid, removed, and the magnesia determined as pyrophosphate.

The filtrate remaining after removing the precipitate by caustic baryta contains the alkalies as sulphates and from this solution the excess of baryta is removed. The filtrate is then evaporated to dryness, dissolved in water with small addition of ammonium oxalate, allowed to stand, filtered, again evaporated in platinum dish and weighed, the residue being sulphates of the alkalies.

IV. The part remaining undecomposed after treatment in III is now treated with a solution of sodium carbonate to remove all soluble silica and this filtrate is evaporated to dryness and filtered and the silica thus obtained added to that

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IV. The part remaining undecomposed after treatment in III is now treated with a solution of sodium carbonate to remove all soluble silica and this filtrate is evaporated to dryness and filtered and the silica thus obtained added to that

obtained from the decomposable iron and this gives the total soluble silica.

V.– The residue after treatment with sodium carbonate is burned and fused with a mixture of sodium nitrate, sodium carbonate & potassium carbonate, removed from the crucible, dissolved in water with addition of acid and the iron, alumina, lime and magnesia determined directly as before.

The high degree of refractoriness of pure alumina and also every dictate of experience has taught us that the more alumina contained in a clay, other things being equal, the higher will be its heat resisting power. Owing, however, to the presence of alkalies and other impurities a clay with a high percentage of alumina may not show as high a degree of heat resistance as one with fewer impurities and less alumina.

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It is to be regretted that no method has yet been discovered by which the alumina of bauxite and other clays can be separated from the iron at a cost low enough to warrant the use of pure alumina in the manufacture of refractory materials. If this could be done successfully and cheaply one great difficulty in the way of this manufacture would be obviated.

In my analyses of clays from this state I have but one in which the per cent of alumina reached forty percent, though others reach very near this high.

The three following clays may be considered, so far as chemical analysis proves, of the finest quality. I and II were analyzed by myself, and III by Mr. A. W. Hare assistant in the laboratory of the School of Mines.

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	I	II	III	
Silica	2.217	0.239	42.437	Decomposed.
Alumina	1.108	8.786	38.740	
Ferric oxide	----	0.110	----	
Ferrous ..	0.665	0.391	0.624	
Lime	0.325	0.968	2.532	
Magnesia	0.198	0.664	---	
Potash	0.084	----	----	
Soda	0.033	0.410	14.953	
Water	10.838	13.300	- -	
Silica	50.629	44.458	---	Undecomposed.
Alumina	33.106	31.606	---	
Ferric oxide	----	----	---	
Lime	0.676	0.349	---	
Magnesia	0.565	0.017		
Totals	100.444	100.398	99.286	

I is the clay used at the Oak Hill works in St. Louis and makes excellent material. II comes from Sullivan on the St. Louis & San Francisco R. R. III is from Ash Grove Missouri. The last two have not yet been used in manufacture. It will be seen from the analyses of these clays that they have a much smaller percentage of alkalies and other impurities than

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some of the best English clays many of which contain as much as two or three per cent of the alkalies alone. There can be but little doubt that the great excellence of the English material comes, not so much from the quality of the clay used as from the methods of manipulation.

By comparing the analyses given with some of the best English clays given below it will be seen that the percentage of alumina in them is much higher than in the English clays, while the percent of alkalies is lower. The best clay used at Dowlais, Wales gives *

Silica		67.12
Alumina		21.18
Ferric oxide		1.85
Lime		0.32
Magnesia		0.84
Potash		2.02
Water	Combined	4.82
	Hygroscopic	1.39
Organic matter		0.90
		100.54

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* Analyzed by E. Riley. Taken from McCreaths Report Penn. Geolog. Survey.

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while the best Stourbridge clay gives*

Silica	63.30
Alumina	23.30
Ferrous oxide	1.80
Lime	0.73
Magnesia	— —
Water	10.30
	99.43

Two other good clays used in the Stourbridge district gave

	A.†	B.‡
Silica	69.25	58.10
Alumina	17.90	26.59
Ferrous oxide	2.97	
Ferric oxide		2.97
Lime	1.30	0.40
Magnesia		0.99
Potash	— — —	1.21
Soda	— — —	— — —
Combined water	7.58	7.57
Hygroscopic "		1.41
Organic matter	— — —	1.21
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In three Missouri clays I found

	I.	II.	III.	
Silica - - -	1.728	1.262	2.468	Decomposed.
Alumina - - - -	3.731	19.658	1.145	
Ferric oxide - - -	0.718	4.416	0.186	
Ferrous .. - -	0.359	0.254	0.503	
Lime - - - - -	0.565	0.189	0.086	
Magnesia - - - -	0.158	0.495	1.305	
Potash - - - -	0.651			
Soda - - - -	0.420			
Water - - - -	8.266	7.752	6.225	
Silica - - - -	51.642	52.451	52.532	Undecomposed.
Alumina - - -	21.878	7.086	30.460	
Ferric oxide - - -	7.278	3.689	3.246	
Lime - - - -	0.905	0.634	1.033	
Magnesia - - -	1.491	0.945	1.045	
	99.792	100.053	100.234	

I is a red clay from Lone Elm Jasper County. II comes from Carterville Jasper Co. III is a white lithomarge from Rolla Phelps Co. and is found between layers of sandstone and magnesian limestone.

By comparison it will be seen that these clays are fully equal

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Magnesia	0.158	0.495	1.305
Potash	0.651		
Soda	0.420		
Water	8.266	7.752	6.225
Silica	51.642	52.451	52.532
Alumina	21.878	7.086	30.460
Ferric oxide	7.278	3.689	3.246
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By comparison it will be seen that these clays are fully equal

to some of the best English clays, and though even these cannot be regarded as our best clays, yet our materials do not equal those of England, hence we infer that the difference of material lies in the difference of treatment.

After the selection of the clay the next important step is the thorough working and kneading of it in order to produce a homogeneous mass. This may be done either by hand or by machine. Hand working has the advantage of being more thorough; machine working is the most rapid and cheap. For the production of some articles, such as muffles, retorts &c, so great is the importance attached to thorough kneading that machine work is discarded entirely. But it seems probable that all necessity for hand work might be discarded by thoroughly pounding the clay by means of a system of

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All fire proof material when made of raw clay alone shrinks considerably when put to use and hence it is found expedient to add to the clay a certain amount of burned material. The quantity of this material is variable and must be determined largely by the quality of the clay used.

In clay whose shrinkage is not very great the amount is generally one fourth of the whole mass. The shrinkage of clay should should be tested previous to use by forming a small cube of known dimensions, heating it, and observing the diminution in size

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After the material has been moulded to the proper form it is finally to be dried, tempered and burned. If the material is firebrick and is made on the ground where used it may in some cases be built into the furnace when dry without previous burning and the expense thus reduced; though, in general, it may be considered the safest plan to burn previou to use. But in the case of muffles, retorts and such articles the long continued tempering is of the utmost importanc

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The famous Stourbridge bricks have a resistance to cracking weight of eleven hundred to fourteen hundred pounds per square inch, and a crushing weight of twenty-two to twenty-four hundred pounds per square inch.

The Dinas brick which contains 98 per cent silica has a cracking weight of eleven hundred pounds and a crushing weight of twelve hundred pounds per square inch.

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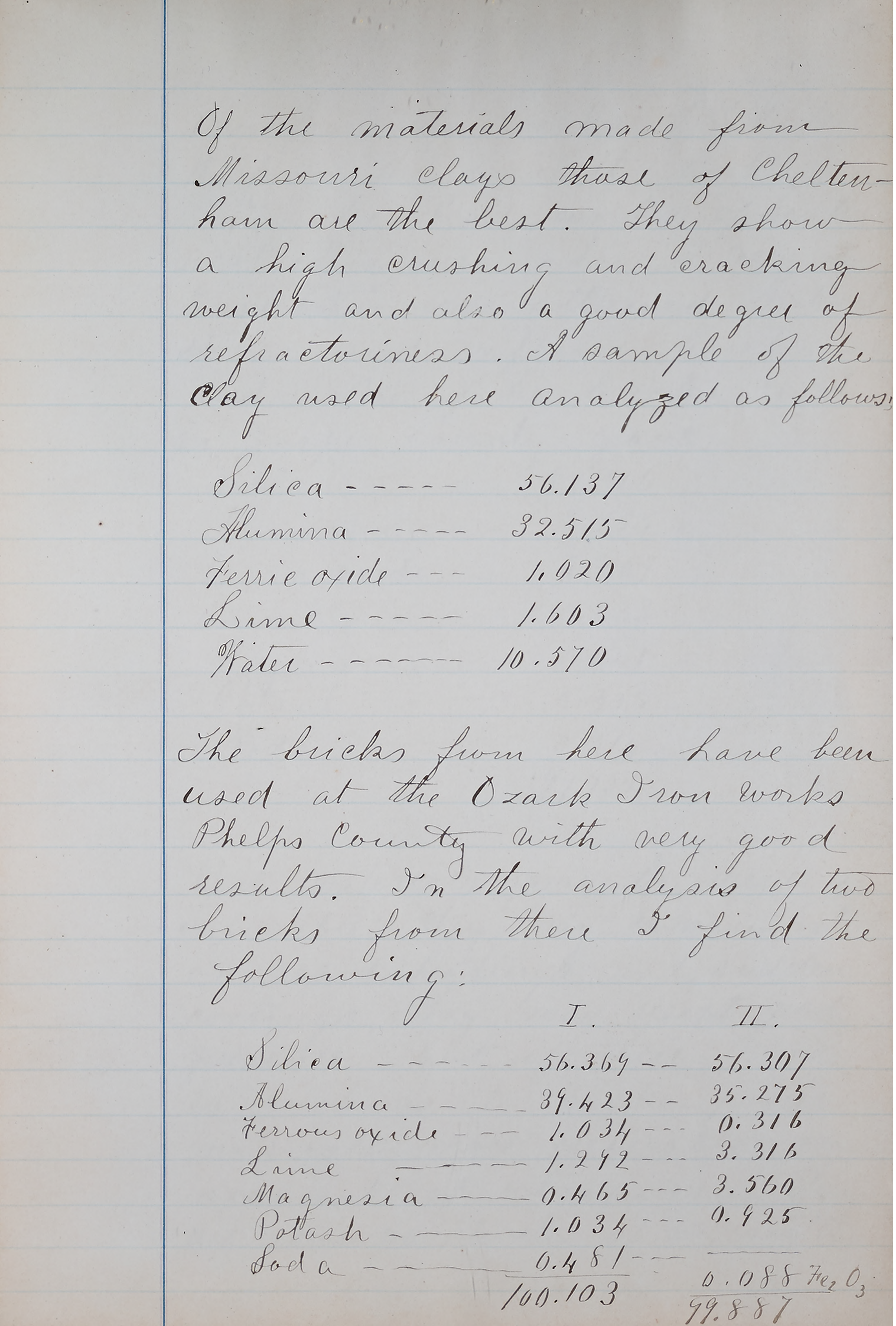

Of the materials made from Missouri clays those of Cheltenham are the best. They show a high crushing and cracking weight and also a good degree of refractoriness. A sample of the clay used here analyzed as follows:

Silica ----	56.137
Alumina ----	32.515
Ferric oxide ---	1.020
Lime ----	1.603
Water ------	10.570

The bricks from here have been used at the Ozark Iron works Phelps County with very good results. In the analysis of two bricks from there I find the following:

	I.	II.
Silica -----	56.369 --	56.307
Alumina -----	39.423 --	35.275
Ferrous oxide ---	1.034 ---	0.316
Lime -----	1.292 ---	3.316
Magnesia -----	0.465 ---	3.560
Potash -----	1.034 ---	0.925
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Silica	83.916
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of labor; that is toward the production of an article equally as good with less labor.

Much labor might be dispensed with in making larger bricks, where bricks are used; in the construction of machines of increased capacity and thoroughness for the kneading of the clay; in the support of but a few manufactories of the largest capacity, giving them an immense custom and thus enabling them to produce their articles at the lowest cost; by locating these establishments only in regions where clay of the best quality can be easily and cheaply mined; and by low rates of transportation.

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the case and where large beds of dolomite exist, as in South Missouri, these might well be utilized. The high degree of resistance of magnesia is well known. Even in contact with melted slags it will resist extremely well. The magnesia may be obtained from the dolomite as follows: burn the stone and then either allow the lime to slake and sift it off or remove it by leaching with water. The magnesia will remain behind. This may then be crushed in a stamp mill and formed into the proper material in connection with small amounts of clay for binding material. This method might be practiced in the absence of highly aluminous clays. Such a material as this might find room for use as bottoms for the Bessemer steel converters. The wear on these bottoms

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is very great; perhaps greater than on any other fire proof material in any other process known. In these bottoms bricks are not often used but the material is put in raw and pounded into shape, being slowly dried. The best record for endurance of these bottoms has probably been made by the North Chicago Rolling Mills. Here the bottoms are made of a mixture of quartz, fireclay and sand which by analysis gives the result below

Silica ---	78.50
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In them I find

	I.	II.	III.	
Silica - - - -	7.320	1.160	1.421	Decomposed
Alumina - - -	4.519	0.907	8.264	
Ferric oxide - - -	4.152	1.081	5.678	
Ferrous oxide - - -	1.424	0.138	0.662	
Lime - - - -	1.988	0.111	0.124	
Magnesia - - - -	2.012	0.097	0.261	
Potash - - - - -	0.551	- -	- -	
Soda - - - - -	0.923	- - -	- -	
Water - ——— -	5.722	2.248	6.699	
Silica - - - - -	53.924	82.206	66.187	Undecomposed
Alumina - - - -	11.014	1.931	2.995	
Ferric oxide - - -	4.454	8.428	6.323	
Lime - - - - —	1.041	1.030	1.218	
Magnesia — - -	0.979	0.437	0.522	
	100.026	99.774	100.354	

I comes from Desloge lead works in St Francis County, II from Minersville, Jasper county and III from Carterville. If not used thus these clays might be made into very good bricks with the addition of a quantity of magnesia obtained as above and in forms adapted to the

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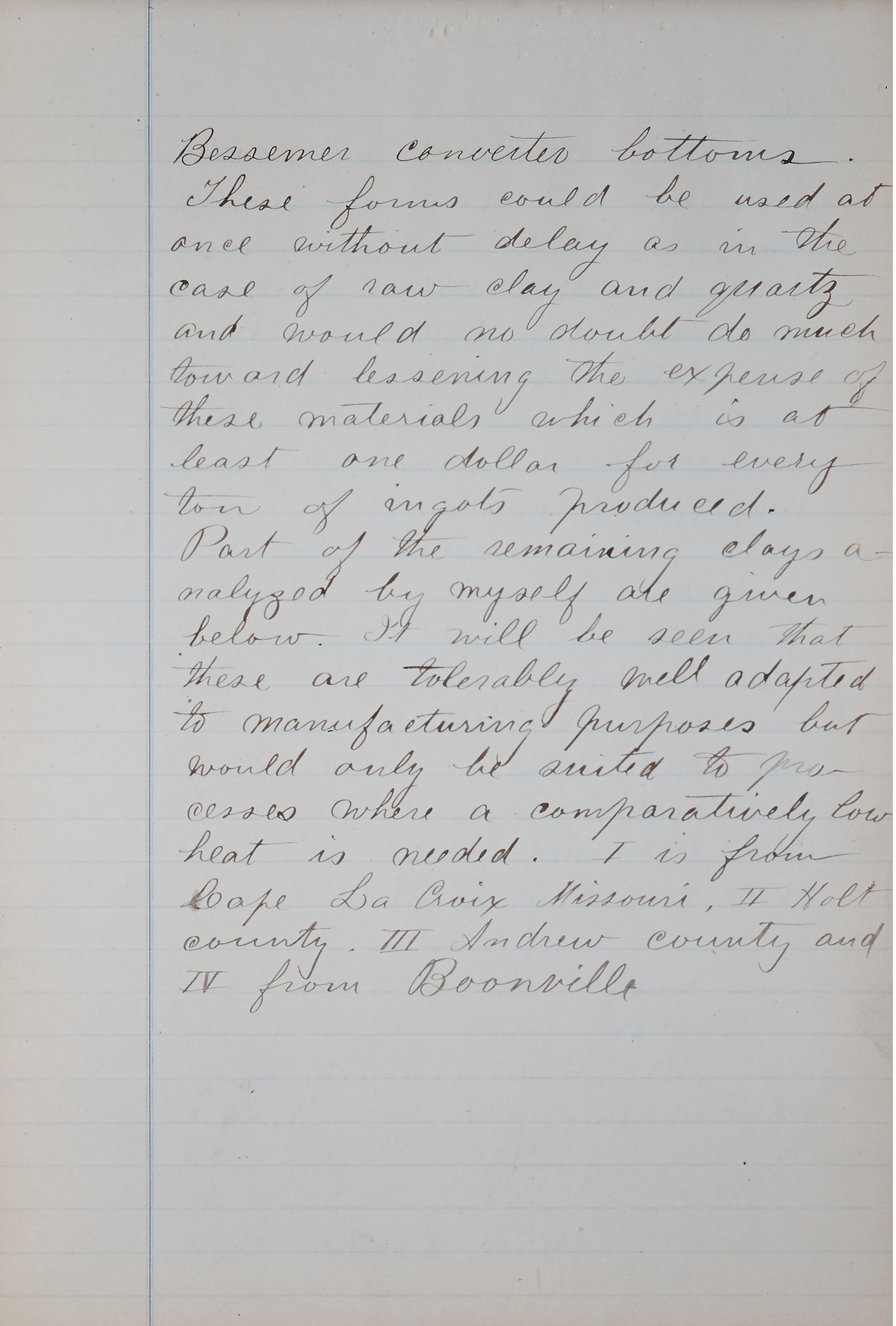

Bessemer converter bottoms. These forms could be used at once without delay as in the case of raw clay and quartz and would no doubt do much toward lessening the expense of these materials which is at least one dollar for every ton of ingots produced. Part of the remaining clays analyzed by myself are given below. It will be seen that these are tolerably well adapted to manufacturing purposes but would only be suited to processes where a comparatively low heat is needed. I is from Cape La Croix Missouri, II Holt county. III Andrew county and IV from Boonville

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	I.	II.	III.	IV.
Silica ———	7.706	3.960	3.242	1.170
Alumina ———	4.129	5.460	11.399	0.495
Ferric oxide ———	0.312	——	0.813	0.289
Ferrous oxide ———	0.128	0.722	1.646	0.176
Lime ———	0.934	6.815	2.700	0.291
Magnesia ———	0.213	0.831	2.161	0.107
Potash ———	3.403	0.409	0.079	1.074
Soda ———	0.487	0.612	0.532	——
Water ———	5.455	11.624	7.657	6.179
Carbonic acid ———	1.046	——	——	——
Sulphur ———	——	——	0.702	——
Sulphuric acid ——	——	——	2.744	2.952
Silica ———	70.748	45.123	56.704	60.730
Alumina ———	4.240	11.717	3.656	23.114
Ferric oxide ——	——	6.538	5.266	2.801
Lime ———	0.934	2.175	0.712	0.461
Magnesia ——	0.192	2.356	0.667	0.353
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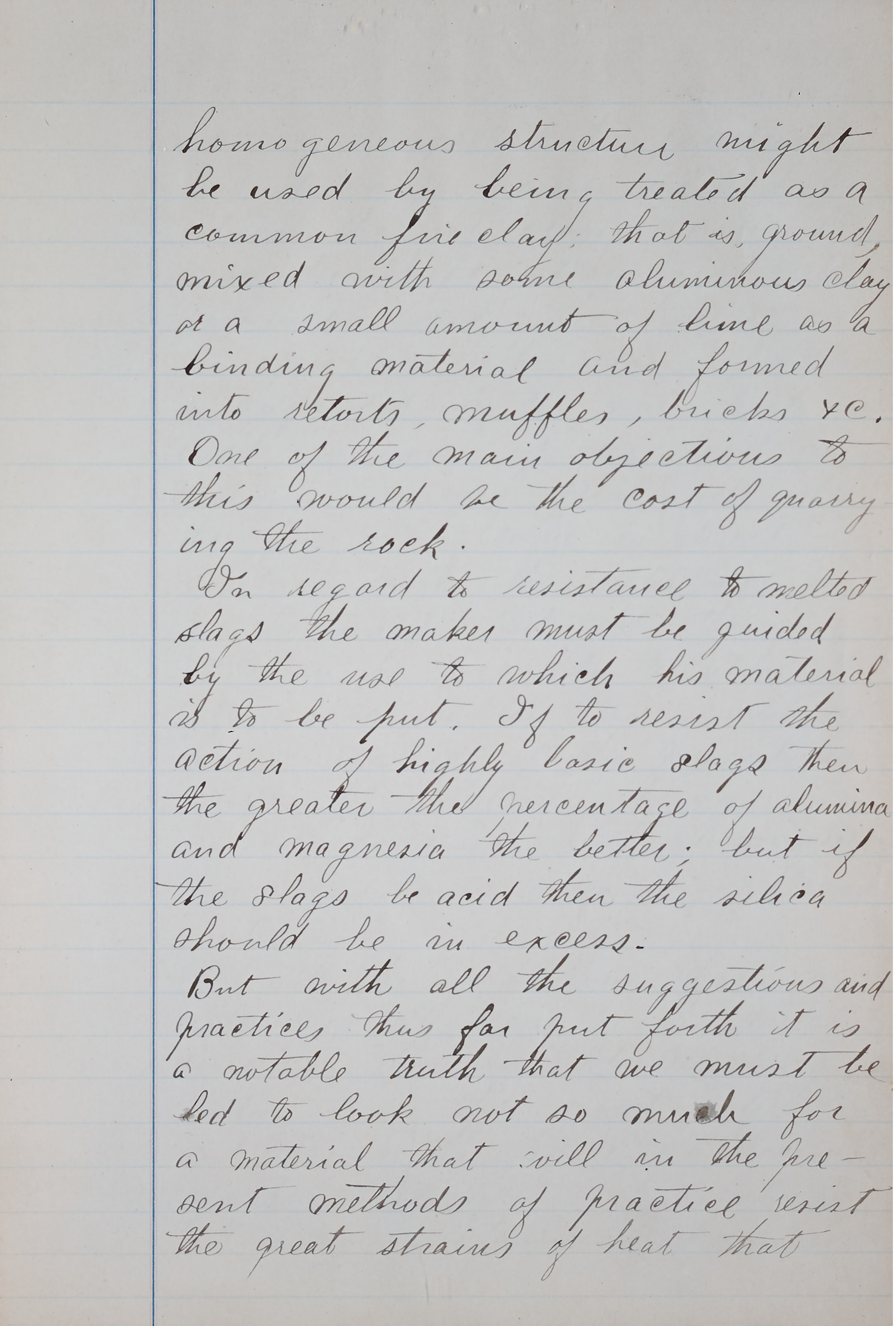

homogeneous structure might be used by being treated as a common fire clay; that is, ground, mixed with some aluminous clay or a small amount of lime as a binding material and formed into retorts, muffles, bricks &c. One of the main objections to this would be the cost of quarrying the rock.

In regard to resistance to melted slags the maker must be guided by the use to which his material is to be put. If to resist the action of highly basic slags then the greater the percentage of alumina and magnesia the better; but if the slags be acid then the silica should be in excess.

But with all the suggestions and practices thus far put forth it is a notable truth that we must be led to look not so much for a material that will in the present methods of practice resist the great strains of heat that

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