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SURVEY OF CERAMIC REFRACTORY PROBLEMS AND MATERIALS SHORTAGES.(U)
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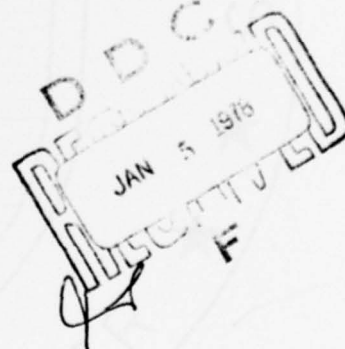
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SURVEY OF CERAMIC REFRACTORY PROBLEMS AND MATERIALS SHORTAGES

GEORGE M. HARRIS and BEVERLY LEES
CERAMICS RESEARCH DIVISION



September 1977

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ABSTRACT

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This report summarizes 171 replies to a questionnaire requesting information on ceramic material shortages and problems with ceramic refractories experienced by metal smelting and metalworking companies. Raw material shortages consist of seven ores; the main problems appear to be with imported materials. Problems range from depletion of the supply to sole source control of quantity.

The problems relating to refractory usage within the metals industries are detailed according to industry and end-item application. Recommendations and possible solutions are included. ↗

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PREFACE

The authors wish to thank the many companies and organizations who responded to their questionnaire and supplied the basic input for this report. It is hoped that the problems outlined in this report, whether material shortages or refractory needs for specific applications, will stimulate efforts by both government agencies and private companies to solve them.

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CERAMIC PROBLEM AREAS

The Army Materials and Mechanics Research Center conducted this survey to uncover areas of Army interest that could be addressed by this Agency or other government installations, and to stimulate efforts within the private sector in areas of commercial interest that may have been overlooked.

This survey was initiated by compiling a list of companies engaged in the metal producing, metalworking, and ceramic refractories industries. Reference sources included Thomas' Register, Poor's Blue Book, and the Product Directory of the Refractories Industry published by the Refractories Institute. The cover letter and questionnaire form are shown in Appendix A.

A total of 659 companies were queried and 171 responses were received. A breakdown of the inquiries into broad industrial categories is given in Table 1. In many cases, inquiries were sent to companies with only peripheral interest or with only a few employees. It was believed that this would give a better overall view of conditions, assuming that the small shop would be the first one cut off from his supply in the event of shortages. Unfortunately, the majority of these companies did not respond.

The response from major producers was excellent. In many cases more than one answer was received from the large corporations and each has been separately evaluated.

For the purpose of this report a ceramic refractory is defined as an inorganic, nonmetallic material that has been fired to high temperature sometime during the course of its manufacture. Both clay and nonclay refractories are included. A refractory must be resistant to thermal shock and withstand abrasive wear and chemical attack in the high heat and other environmental factors to which it is exposed. End-item applications include the normal, recognized items such as bricks, refractory cements (gunning mixes, plastic mixes, dry) used to form monolithic structures, nozzles, troughs, pump components for molten metal

Table 1. DISTRIBUTION OF INQUIRIES

Category	Inquiries	Replies	%
Aircraft	145	34	23
Refractories	128	31	24
Ferrous Metals	125	22	18
Nonferrous Metals	69	20	29
Ceramic Tool Bits	31	4	13
Automotive	21	5	24
Glass	19	4	21
Ingots	18	5	28
Abrasives	17	16	94
Ceramics	18	5	28
Metals	16	0	0
Raw Materials	16	14	88
Kilns, Furnaces	16	3	19
Electrical	14	7	50
Smelting	4	0	0
Associations & Societies	2	1	50
Total	659	171	26

usage, fusion cast blocks, etc. In addition, ceramic tool bits, ceramic dies, and ceramic tooling for fabrication of metal parts at elevated temperatures have been included.

According to the Bureau of Census, the total value of the refractories produced in 1974 was in excess of one billion dollars. Figure 1 illustrates the domination of the iron and steel industries in the use of refractories, from 56.7% to 59.8% of the market during the 1972-1974 time period. Nonferrous metals account for only 6.0% to 6.8% of the reported refractories shipments.

It can be assumed that the major refractory producers conduct most of their R&D efforts toward improvement of refractories to yield the greatest dollar return on investment. However, it cannot be assumed that R&D projects are in progress for all types of refractories used in iron and steel plants. In general, the major ceramic refractory producers do not supply products across-the-board, especially for specialties such as basic brick, chrome ore, crucibles, fused castings, ladle brick, semi-silica brick, silicon carbide brick, zircon brick, and zirconia brick. These, and specialty shapes like nozzles or pouring spouts, are generally supplied by the smaller refractory companies and one or two of the major firms. The small companies that operate on a job-shop basis cannot afford the luxury of an R&D laboratory. The large manufacturer cannot be expected to expend major efforts to improve the performance of low volume items where the dollar return would be minimal. It is in this area of specialized refractories that major breakthroughs in product improvement can probably be achieved.

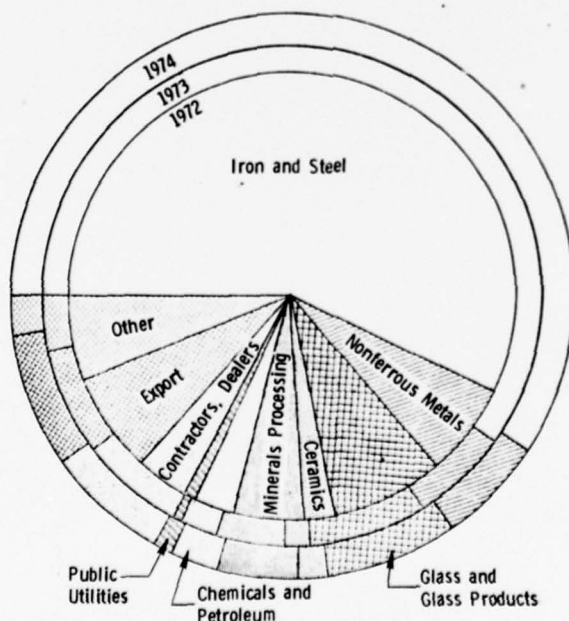


Figure 1. Reported refractories shipments by markets, 1972 through 1974 (millions of dollars). Source: "Energy Utilization in the Refractories Industry, 1972-1974," Battelle Columbus Laboratories.

The list of problem areas is too extensive to discuss individually in this section. Each problem area is presented in Appendix B along with a proposed solution or probable technical projects which could alleviate the situation. The numbers listed in Appendix B relate to the problem area numbers shown in Table 2. A few of the entries are not related to the metals or metalworking industry, but they were submitted in answer to the questionnaire and appear to be legitimate problem areas. The majority of suggested solutions were submitted by companies having those problems; the accuracy or technical feasibility of their recommendations have not, in every case, been verified.

Table 2. PROBLEM AREAS

CATEGORY	WHERE USED	PROBLEM AREA NUMBER
FERROUS METALS	SLIDING GATE LADLE VALVES	1
ALUMINUM	CHARGING WELL ARCHES,	2
	RECLAMATION FURNACES	3
	REVERBERATORY FURNACE DOOR	4
	HOLDING AND MELTING FURNACES	5
	REMELT FURNACE	6
	LAUNDERS	7
	PROTECTION TUBES AND HEATING ELEMENTS	8-10
ZINC	SMELTING FURNACE	11
	REFRATORIES FOR CONTINUOUS OPERATION	12
	REFRACTORY LINED LADLES, ETC.	13
	THERMOCOUPLE WELLS	14
	HEAT EXCHANGERS	15
	PNEUMATIC DUST CONVEYORS	16
	MIXER BLADES	17
	COATINGS ON STEEL	18
	INDUCTION MELTING	19
LEAD	LEAD SMELTERS	20
TITANIUM, HOT FORMING	OXIDATION RETARDANTS	21-26
	DIE MATERIALS	27
MOLY-BASE ALLOYS	INVESTMENT CASTING	28, 29
REFRACTORY METALS	EXTRUSION DIES	30-38
CERAMIC CUTTING TOOLS	METAL MACHINING	39
PUMPS	MOLTEN ALUMINUM	40
	MOLTEN ZINC	41
GENERAL REFRACTORIES	SINTER W BILLETS IN H ₂	42
	LOW TEMPERATURE FURNACES, 1700 F	43
	STEEL CHIMNEYS	44
	HIGH TEMPERATURE FURNACES, HOSTILE ENVIRONMENTS	45
	FUSION CASTING	46
	SUPERALLOYS	47
	TROUGH LININGS	48
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	IRON CORELESS FURNACE LININGS	50-68
	HOT DIE FORGINGS, GLASS, WC VACUUM FURNACES	
MISCELLANEOUS REFRACTORY PROBLEMS		

Ferrous Metals: As previously stated, the iron and steel industries purchase more than fifty percent (dollar value) of all refractories produced in the United States. The majority of these products are high volume items manufactured by the major refractory companies. These companies have extensive R&D facilities and work closely with their customers, many of whom have their own ceramic engineering departments and/or ceramics development laboratories. In all probability, their major projects relate to the standard refractories used in smelting and reheat furnaces, hot metal cars, ladles, etc., where an extension of refractory life provides a significant cost savings to the user in reduced down-time and maintenance.

Yet, there are other problem areas that are probably receiving little or no attention, mainly because the refractories are specialized products usually supplied by the smaller manufacturers or because they do not command sufficient dollar sales volume to warrant more than a cursory investigation. Included in this listing are sliding gate ladle valves, nozzles, pour-spouts, troughs, etc. Several of these are reported in Appendix B. It is recommended that efforts be initiated to improve the thermal shock resistance, abrasion and corrosion resistance, and spalling resistance of these materials.

Aluminum: Only a few aluminum companies responded to the questionnaire, but each noted several problem areas, and in some cases proposed solutions. A number of replies focused on the need for improved thermal insulation, a vital necessity in this age of escalating fuel costs and genuine concern for energy conservation. Other refractory improvements of major interest to the aluminum industry are in thermal shock resistance, corrosion, and compatibility with hostile environments. Generalized problems and some solutions are listed below; more detailed information is contained in Appendix B.

(1) Excessive heat losses in holding and melting furnaces shorten furnace life and waste energy. (2) The insulation used on reverberatory furnace doors should be improved to provide thermal shock resistance; the inclusion of ceramic fibers in the castable may retard thermal shock. (3) The refractory brick or cast refractory in charging wall arches is subject to spalling; an R&D project should investigate the formation of nephelite and determine if this is the cause of failure. (4) The refractories used in the air recirculation system of remelt furnaces are attacked by fluxes added to the melt; an R&D project should be started to identify the reactant phases and means of prevention. (5) Steel troughs (coated with a ceramic wash) used to transport molten aluminum have short life and high thermal conductivity, necessitating the use of torches to prevent solidification of the metal; substitution of a ceramic would reduce thermal conductivity and prolong life. (6) Silicon carbide protection tubes for nichrome heating elements used to melt aluminum have short life; a project to determine the feasibility of substituting another ceramic for silicon carbide should be started.

Zinc: Only one company from the zinc industry responded to the questionnaire, but they submitted a list of ten problem areas and these are exactly the type of "nitty-gritty" problems which were sought. Comment: If these problems are common throughout the zinc smelting industry, it would appear that they are in need of major assistance from the refractory manufacturers to develop suitable

materials for their specialized usage. (1) Super-duty brick currently used in electrothermic zinc melting furnaces are not sufficiently resistant to high temperature fluxing and reduction by coke or zinc oxide-containing sintered feed materials. (2) In the same category is the lack of a sleeve (ceramic) to encase the graphite electrodes (12 in. dia.) to prevent corrosive deterioration of the furnace lining immediately beneath the electrodes. (3) A substitute, long lasting material is required in the "curtain" of electrothermic smelting furnaces at temperatures of 1200 C in a strongly reducing and occasionally oxidizing atmosphere. (4) A refractory is needed which will resist chemical attack from oxidizing zinc vapors at 1900 C to 2000 C. Alumina or magnesia refractories, presently used, have the temperature capability but lack corrosion resistance. (5) Improved castables are needed for refractory linings in ladles, troughs, and tubes which fail due to thermal shock and/or corrosive attack from molten zinc. (6) A corrosion-abrasion resistant coating is needed to lengthen the life of steel used in conveyor-feeder systems, heat exchangers and as mixer blades which are extensively used throughout the zinc industry.

Lead: Only one reply was received from a lead company, but it presents a major problem. Evidently the lead industry has several processes for the direct conversion of lead from lead sulfide. However, there is no ceramic refractory on the market which is chemically inert to both the sulfide and molten metal at processing temperatures. It appears that a project is warranted to investigate the various reactions and identify the reactant phases unless this work has already been done, in which case an R&D project could be started to develop the refractory which would remain stable under these conditions.

Titanium, Hot Forming: Problems associated with the hot forming of titanium are common throughout the aircraft industry. Five separate companies stated their problems with the tooling for hot forming titanium to shape, for hot extrusion dies and hot metal platens. The hot-forming-to-shape tooling for the fabrication of curved or contoured parts is generally a castable ceramic, usually fused silica for its thermal shock resistance and low coefficient of expansion. The main problems are the low tensile strength, mismatch in thermal expansion between the ceramic and titanium, poor surface finish and the relatively high coefficient of friction of the ceramic which restricts the normal expansion and contraction of the titanium. In addition, one complaint was registered against a ceramic supplier for the bag-to-bag inconsistency of the castable material. Evidently the material varies in particle size, particle size distribution, and contains a cement of varying quality. These lead to wide variations in the setting time, cold crushing strength, and die surface hardness of the ceramic tooling. Other needed improvements in ceramic tooling include the repair or modification of existing tooling. Current ceramic materials are difficult to repair or modify once they are cast. The aircraft companies are also looking for a ceramic with increased temperature capabilities up to 1093 C as compared to the present 982 C requirements, as well as longer die life. Separate programs to improve the various properties of ceramic tooling should be initiated.

Metal dies are used for the hot extrusion of titanium and refractory alloys. Die wear is excessive; they must be refurbished after a few runs. A feasibility project to determine the suitability of ceramic dies, possibly of silicon nitride, silicon carbide, or tungsten carbide should be funded.

Inconel 802 heated platens are used for hot forming titanium. However, the Inconel tends to warp and deform after extended use under high forming pressures at elevated temperatures. A program should be undertaken to develop a ceramic suitable for this usage.

Molybdenum-Base Alloys: Ceramic investment casting molds are used for skull melting and casting molybdenum-base alloy turbine wheels and blade rings for automobile and truck turbine engines. Temperature requirements are in the 1510 C range. The ceramic investment mold material is unsuitable because it tends to react with carbon or the alloying elements. Castings of TZM have been possible with proprietary ceramic mixes. However, it has still not been possible to cast molybdenum alloyed with hafnium and carbon because of the reaction between the carbon and the mold material. The company that submitted this problem suggested that the solution may lie with a ceramic which has no phase transformation or reaction with carbon in the 1510 C temperature range; SiC and HfC composites should be considered.

Extrusion Dies: Extrusion of refractory metals, titanium, superalloys, and stainless steel is limited and expensive due to poor wear resistance and short die life. Dies may be of steel, H-11 or H-13, or of a ceramic material such as zirconia. Generally, the ceramic is used as a facing or insert due to low tensile strength. There is a definite need for an improved ceramic die and HfO₂ is a recommended candidate material. Both zirconia and hafnia must be stabilized to prevent crystalline inversions which cause discontinuities in the thermal expansion, accompanied by volume changes of sufficient magnitude to fracture the oxide body.

Glass is often used as a high temperature lubricant in hot extrusion of metals. Die life can also be improved by development of a more compatible lubricant. The overall problem can be approached by R&D efforts in both directions, improving the die material or discovering a more compatible glass lubricant, or by simultaneously addressing both.

Ceramic Cutting Tools: There were ten replies listing ceramic cutting tools as a major problem area in metal machining. The main criticisms of ceramic cutting tools appear to be: (1) low impact resistance; (2) inability to make interrupted cuts; (3) lack of ductility; (4) low transverse rupture strength; (5) lack of microstructural homogeneity of composite tool bits; and (6) low vibration resistance.

There is no doubt that additional R&D efforts to improve ceramic tool bit performance would be beneficial, particularly in the development of a specific tool bit for the machining of a specific ferrous or nonferrous metal. However, the limitations of ceramics, i.e., lack of ductility, must be recognized. Ceramics do not behave like metals in tool bit application. Ceramics have limitations; machinists should operate within the framework of these parameters and not expect ceramics to fulfill the requirements of the all-purpose tool bit. In the meantime, research on ceramic tool bits can have the objective of improvement at a gradual rate to provide increased performance over materials now in existence.

Pumps for Molten Metals: This category extends across the board for the transportation of both ferrous and nonferrous metals. The specific problem areas reported concern the need for high volume, portable pumps for use with molten aluminum and molten zinc. For aluminum, small pumps are already available but their size limits the use to quality control samples. It was reported that one aluminum company was working on the development of a large pump, approximately three feet in diameter. No details are available.

The complaints against pumps for molten zinc are the excessive maintenance and short life of the parts in contact with the metal. A more rugged pump with greater capacity would reduce the cost of transporting molten zinc.

It would appear that the pump manufacturers and specialty refractory manufacturers should get together and jointly attack this problem. It does not sound like an easy solution is forthcoming as erosion due to the rapid flow of metal, thermal shock, design configuration limitations, chemical reactions, vibration, etc., are problems to consider.

Refractories in General: This "catch-all" category runs the gamut from refractories for fusion casting at 2300 C in hydrogen atmosphere to refractory insulation for operation in air at 930 C. In some cases, a substitution of off-the-shelf refractory materials appears to be feasible while other solutions require extensive R&D programs.

OSHA Restrictions: Replies to the survey only brought three responses where OSHA restrictions are of concern. Two of them referred to the use of asbestos: (1) a machinable board containing asbestos where the temperature requirement is 1095 C and the recommendation is for mullite fiber to be used as a replacement and (2) where asbestos downspouts are used in the aluminum reverberating furnace.

The third item concerns the use of silica. While this material is not presently banned by OSHA, there is growing concern that it may be on OSHA's list in the future due to the danger of silicosis. This material is used extensively in iron coreless furnaces and in countless other applications where castable silica cements and gunning mixes are used. However, the majority of these mixes are alumina based and these should not be affected by any future OSHA action.

CERAMIC MATERIALS SHORTAGES

The materials shortages listed in this section have been supplied by ceramic companies in response to our questionnaire. Although limited in scope, the list does include low quantity materials and others not on the national stockpile list. Shortages may exist for a number of reasons: (1) short supply; (2) limited production; (3) increased demand; (4) fuel shortages; (5) slow deliveries; (6) curtailment of imports; and (7) environmental protection requirements. Whatever the reasons, attempts must be made to relieve the shortages by appropriate means. This can range from increased production, substitution of a lower grade domestic supply, development of a substitute material through research and development efforts, or improved manufacturing technology to meet OSHA requirements.

There are numerous reference sources for information on material and energy shortages.¹⁻⁹ Of particular interest in the area of materials shortages are the excellent bibliographies contained in the paper by Edward Dyckman which was presented at the DoD Materials Shortages Workshop² and AMMRC Report MS 75-8 on metallic materials.⁹ The General Services Report, "Strategic and Critical Materials: Descriptive Data"⁵ is a listing of strategic and critical materials, together with geographic origins and possible substitutes. However, it must be remembered that in the case of stockpiled ceramic materials, raw materials alone are considered. The present survey also includes manufactured items in short supply. Refractory manufacturers may list an ore as being in short supply and this could result in the shortage of a particular grade of refractory brick used, for example, in a smelting furnace for the reduction of a metallic ore. A domino effect will develop as the ceramic supplier will list the ore as a shortage item, while the refractory manufacturer and smelter will list the refractory brick. Or, even though there may be a suitable supply of metallic ore, delivery delays of six months or a year could be encountered because of shortages in furnaces, linings, or equipment.

This listing is admittedly incomplete, but it demonstrates the realm of foreseen shortages within the ceramic industry. Also, funding and time constraints did not permit a check as to the completeness of the responses; for example, in some cases a suitable material or substitute may, in fact, be commercially available without the user being aware of its existence. In all probability this is not likely, but may be a possibility and should not be completely ignored.

MATERIALS SHORTAGES

Mullite: There is a lack of high purity sintered mullite for use in refractories for specialized applications. For example, refractories in furnaces requiring controlled reducing atmospheres and low dew point cannot tolerate ferric oxide and titania impurities which are unstable in reducing atmospheres. Yet commercial grades of calcined kyanite and bauxite contain these impurities. High purity refractories are also needed for induction melting and/or heating. According to one company, commercially available high purity refractories are not available for these and similar applications.

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9. ABBOTT, K. H. *Army Utilization Management of Metallic Materials*. Army Materials and Mechanics Research Center, AMMRC MS 75-8, October 1975.

Bauxite: The major complaint with bauxite used in the refractories industry appears to be the escalating cost and limited availability of the calcined material from South America. One manufacturer mentioned that a percentage of this bauxite has been replaced by calcined domestic grade bauxite. Another suggested that domestic minerals be upgraded to the same composition and refractoriness of the imported bauxite. For general refractories the requirement is: greater than 85% alumina with less than 5% iron oxide plus titania.

Chrome Ores: The concern of manufacturers of chrome refractories is two-fold: (1) the depletion of existing supplies within the free world and (2) the increasing dependence upon sources under cartel control. The Philippine Islands supply the bulk of the refractory grade chromite ore, with Turkey and the Republic of South Africa as secondary sources. One suggestion for improving the situation was the recommendation that a project be initiated to study the feasibility of substituting dead-burned dolomite for chrome oxide in refractories. The effect of this substitution on refractories for the ferrous, nonferrous, cement, lime-producing and glass industries should be evaluated.

Magnesite: The major problem appears to be one of fluctuation within the market. The magnesite basic brick is predominantly used in the steel industry, and when steel production is at a peak, delivery time is lengthy. One user stated that, at present, basic brick delivery is approximately one year.

Iron Oxide (Fe_2O_3): The chief source of iron oxide used in producing ceramic permanent magnets is a by-product of the steel industry; it is regenerated from steelmaking "pickle liquor." The shortage exists because of the limited number of regeneration plants in operation. These magnets are utilized in loudspeakers, D.C. motors, separators, magnetos, microwave ovens, etc. One magnet manufacturer stated that one way to increase the supply of iron oxide is to tighten the enforcement of ecology regulations and prevent steel mills from dumping pickle liquor into deep wells. It is not clear whether this manufacturer is suggesting that existing regulations be enforced or more stringent regulations be instituted. If the latter, this is the only suggestion implying that OSHA or EPA are too lenient with their regulations.

Silicon Carbide: This material is used primarily for abrasives or as refractories for specialized applications. The main problem appears to be limited production facilities which, despite sufficient supply, does not allow for industrial expansion. In addition, there is a potential shortage of silicon carbide heating elements. The reason is that only 5 tons per 100 tons produced is of sufficient purity for heating element usage. According to one manufacturer, costs have escalated approximately 300% in the past five years.

Graphite: Evidently there is a serious problem with a particular grade: natural flake crystalline graphite. The principal source of this material is the island of Madagascar and supply is sporadic at best. The natural flake graphite is required for stopper heads used in pouring and controlling the flow of steel for both ingots and continuous casting. Apparently other forms of graphite cannot be used for this application.

DISCUSSION AND RECOMMENDATIONS

In attempting to relieve material shortages there are generally several approaches to the problem. In most cases, economic factors play an important part. However, it must be remembered that economic conditions change with time and what was considered uneconomical a few years ago may be perfectly justifiable in today's or tomorrow's marketplace. As costs of raw materials spiral upward it often becomes feasible to reinvestigate the use of a substitute material, even though this identical solution was rejected in the past. The same holds true for other solutions such as: refining existing ores to match the more stringent specifications of the imported material now in use; or by expanding manufacturing facilities to meet current and future demand.

Beyond the economic logic for accepting or rejecting a project for relieving material shortages, other considerations may be of equal or greater importance. For example, consider natural flake graphite. Is Madagascar the only acceptable source? Is this the only satisfactory material that can be used for stopper heads and similar applications in the steel industry? If so, then efforts should be undertaken to provide either an alternate supply or a substitute material even if the cost is higher. This will at least provide an alternate source in case the supply is cut off due to political or logistical reasons.

Mullite: To improve the refractoriness of mullite ($3Al_2O_3 \cdot 2SiO_2$), ferric oxide and titania impurity levels must be reduced. If this is done, it appears that there is a market for a specialized refractory used in hydrogen atmosphere furnaces and for induction heating. The feasibility of reducing these impurities in mullite should be determined.

Bauxite: Domestic grades of bauxite contain a higher percentage of iron oxide and titania than calcined South American bauxite. As the cost of the South American material is increasing and the quantity diminishes, the time may be right to consider funding research to reduce impurity levels in domestic bauxite to match the specifications of that imported. Also suggested is an R&D project to determine the amount of domestic material that can be substituted for South American bauxite without significantly affecting the properties of the manufactured refractory brick.

Chrome Ores: A project should be considered to determine the availability of a substitute material for chrome ore used by the refractory industry in chrome-magnesite and magnesite-chrome brick. One recommended approach is to substitute dead-burned dolomite for the chrome. While the dolomite is a solid solution of calcium and magnesium carbonates, it has been used successfully in repairing and patching furnaces composed of magnesite brick.

Graphite: There can be three approaches to solving the problem associated with the natural flake crystalline graphite found on Madagascar: (1) determine the suitability of domestic source even if the material is slightly more costly; (2) attempt to synthesize the material; and (3) attempt to develop a substitute form of graphite or other material. If this graphite problem is as critical as stated (that this is the only material suitable for stopper heads in the steel industry), it requires attention at an early date.

Silicon Carbide: At present only 5% of the silicon carbide produced is of sufficient purity for use as heating elements. There appears to be an impending shortage of these heating elements and it would seem logical to assume that manufacturers would concentrate their efforts on improvements in processing technology to increase the yield of heating element grade material per ton of silicon carbide produced.

Iron Oxide: The information furnished is too sketchy to make any recommendations.

PROPOSED ARMY PROJECTS

The following problems are proposed as possible candidates for research or procurement funded projects. It is suggested that specific project proposals be prepared to solicit funding for these efforts.

Ceramic Tooling: Ceramic tooling is utilized throughout the aircraft industry for the hot forming of metallic (titanium) structural members. Improvements in tooling life and surface finish are needed to reduce the cost of the formed sections. It is evident that thermal cycling of the fused silica tooling produces cristobalite crystallization (a form of crystalline quartz). This causes cracks within the tooling structure and leads to structural failure after a limited number of thermal cycles. Reduction of impurities (especially alkalis) should retard cristobalite formation and increase tooling life. The rough surface finish prevents normal expansion and contraction of the metal section during the thermal cycle and puts undue stress on the ceramic tooling. Improvement of surface finish by the application of a fused silica glass layer should reduce the coefficient of friction to a workable level.

Graphite: Evidently the entire steel industry relies on a unique form of graphite, natural flake, for stopper heads; no satisfactory substitute material has been found. The island of Madagascar is the sole source and shipments are made on an irregular basis. A program should be implemented to produce a substitute material, locate a satisfactory second source, or develop a synthetic form of graphite with the unique properties of the natural flake material. A preliminary survey of the steel industry and graphite suppliers would be warranted before undertaking this project to determine the actual requirement for this specific graphite. The project's priority could then be reasonably established.

Refractories for Zinc Smelters: Evidently there are several sections of zinc smelters where refractory wear is excessive. A project should be started to determine the cause of failure - erosion, chemical attack, spalling, etc. Once the failure mode has been determined, efforts can be made to either replace the refractories with an improved off-the-shelf item to withstand the hostile environmental conditions or modify the existing refractories' compositions to upgrade their performances.

Hot Forming Titanium: This project would attempt to solve the problems presently encountered in the hot extrusion and hot stamping of titanium shapes.

For hot extrusion both ceramic diffusion-bonded coatings and all-ceramic dies should be evaluated. All-ceramic dies would probably be superior to coated dies provided the ceramic is in compression, not tension. Ceramic-coated platens for hot forming of titanium shapes by pressing should be evaluated as they would provide increased hardness and wear resistance over steel die materials.

Ceramic Tool Bits: It is extremely doubtful that ceramic tool bits will ever be perfected to withstand interrupted cuts, chattering, excessive vibration, and the like. This is due to the lack of ductility inherent in the material. However, this does not mean that efforts should not be undertaken to improve their performance. Ceramic coatings on metals and higher hardness ceramic materials are two approaches to improvement in performance of ceramic tool bits. A literature survey would be beneficial to determine whether or not exploratory work has been done in the area of ceramic tool bit development for the machining of specific metals. For example, which ceramic works best on low carbon steel, cast iron, nickel, etc? Particular attention should be given to the test equipment; it must be sufficiently true and instrumented for pressure, feed rate, etc., so that results are reproducible and any failure of the tool bit is the result of the material and not the machine.

APPENDIX A. SURVEY COVER LETTER AND QUESTIONNAIRE FORM



DRXMR-EO

DEPARTMENT OF THE ARMY
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
WATERTOWN, MASSACHUSETTS 02172

The Army Materials and Mechanics Research Center is conducting a survey on the need for substitute or improved ceramics for use by the metalworking industry. This information will be used in formulating future planning of both applied research and production oriented projects.

For the survey, we are interested in all possible ceramic applications in metalworking. This includes, but is not limited to, ceramic refractories for furnace linings, ladles, nozzles, pouring spouts, molds, hot forming dies, and tool bits. There is also the possibility that ceramics could be utilized in applications where metals are currently being used and the metallic state-of-the-art is being pushed to the limit due to requirements for high temperature, abrasion resistance, etc.

Of equal importance to us are problems resulting from material shortages where substitute materials must be found to replace critical ones. In the same category is the problem arising when a material becomes too costly and a more cost effective substitute must be found to insure that the end item remains competitive in the marketplace.

For your convenience we are inclosing a form which contains the basic information for our use. Please feel free to add to it in any way you wish and use additional pages if desired.

It is our intention to use the information gathered on this survey for internal purposes only. Existing Army regulations will be used to protect proprietary rights, so proprietary information supplied should be so identified.

We want to thank you for your cooperation.

Sincerely,

1 Incl
as

GEORGE M. HARRIS
Ceramic Engineer
(617) 923-3258

SURVEY OF CERAMIC PROBLEM AREAS AND MATERIAL SHORTAGES
ASSOCIATED WITH METALWORKING INDUSTRIES

COMPANY:

ADDRESS:

PROBLEM:

MATERIAL NOW USED:

POSSIBLE SOLUTION:

DoD OR OTHER FEDERAL AGENCY PURCHASED COMPONENT OR SYSTEM AFFECTED:

COMMENTS:

SIGNATURE (optional)

Incl 1

APPENDIX B. LISTING OF PROBLEM AREAS REPORTED BY THE METALWORKING INDUSTRIES

Ferrous Metals:

1. *Problem:* There is excessive erosion of the high alumina (70% +) refractory plates comprising the sliding gate ladle valves in steel furnaces.

Proposed Solution: Develop basic refractory plates (high MgO) with increased erosion resistance to the molten metal.

Aluminum:

2. *Problem:* In the aluminum industry the insulation for reverb furnace doors is refractory castable materials. The doors are heavy and subject to thermal shock.

Proposed Solution: Develop a fibrous ceramic insulation to provide a lighter weight door with improved insulation and thermal shock properties.

3. *Problem:* The charging well arches in aluminum reclamation melting furnaces are presently made with either conventional high alumina (85% to 90%) burned brick, basic (chrome-magnesite) brick or high alumina cast refractories. All fail as the result of spalling; nephelite is produced, but how this ties in with the failure is not known.

Proposed Solution: R&D to isolate the problem of reactions taking place, followed by development of refractory to meet known conditions.

4. *Problem:* Excessive heat loss in aluminum holding and melting furnaces. Attempts have been made to use insulated sheets near the outside wall but cracks usually form in these refractories, allowing molten metal to leak through.

Proposed Solution: Develop refractory material with $K \leq 1$ (similar to Kaowool or Fibrefrax) which does not wet aluminum or coat with low thermal conductivity coating which is nonwetting.

5. *Problem:* In the aluminum remelt furnace corrosive fluxes are added which affect the recovery or recirculating system.

Proposed Solution: Develop a refractory impervious to the furnace conditions.

6. *Problem:* Launderers (troughs) made of steel coated with a ceramic wash are presently used for handling molten aluminum. They have a short life and high thermal conductivity, so torches must be used to supply lost heat.

Proposed Solution: Develop a ceramic with low thermal conductivity that is nonwetting to molten aluminum.

7. *Problem:* Silicon carbide is presently used as protection tubes for heating elements used to melt aluminum, nichrome wire being used as the heating element. These elements are dipped into molten aluminum and have a relatively short life.

Proposed Solution: Develop ceramic with nonwetting, high thermal conductivity and good mechanical properties at 705 C. The electric immersion heaters would be widely used for melting aluminum, offering four times the efficiency of fuel-fired furnaces if protection tubes and heating elements had longer life.

Zinc:

8. *Problem:* Super-duty firebrick are not satisfactory for use in zinc smelting furnaces.

Proposed Solution: Develop low cost refractory resistant to high temperature fluxing and reduction by coke and zinc oxide.

9. *Problem:* A cylindrically shaped ceramic is needed to fit around the 12"-diameter graphite electrodes to protect the electrothermic zinc furnace lining directly under the electrodes from corrosive attack.

Proposed Solution: Develop a cylindrical refractory ceramic which will resist high temperature fluxing and reduction by coke and zinc oxide.

10. *Problem:* A durable refractory is needed to replace graphite in the "curtain" of electrothermic zinc smelting furnaces.

Proposed Solution: Develop a refractory ceramic with good thermal shock resistance and low electrical conductivity which can withstand a strongly reducing, but occasionally oxidizing, environment at temperatures of 1200 C.

11. *Problem:* A suitable refractory is needed which will not react with zinc oxide at temperatures of 1900 to 2000 C. This will replace alumina or magnesia refractories which have the temperature capabilities but react with zinc oxide to form lower melting compounds. The improved refractory would permit improved burners used to oxidize zinc vapor to produce zinc oxide.

Proposed Solution: Develop refractory to meet these specific requirements.

12. *Problem:* Commercially available castable refractories are unsatisfactory in lined ladles, troughs, and tubes used in contact with molten zinc. Deficiencies are in thermal shock and contamination.

Proposed Solution: Develop improved castable refractory which does not undergo phase transformation or react with zinc.

13. *Problem:* Unsatisfactory thermocouple wells exposed to zinc liquid and vapor at temperatures up to 950 C.

Proposed Solution: Same as #12.

14. *Problem:* Steel cooling tubes presently used in heat exchangers for cooling molten zinc are corroded by the metal, causing water leaks which constitute a safety hazard.

Proposed Solution: Either develop a satisfactory coating for the steel tubing or substitute a suitable refractory ceramic.

15. *Problem:* Excessive wear and high maintenance are experienced on valves and hoppers of pneumatic dust conveyor-feeding systems in zinc plants.

Proposed Solution: Substitute wear-resistant ceramic for steel in the critical areas of feeding-conveyor systems.

16. *Problem:* Present stainless steel turbine-type mixer blades are unsatisfactory for alloying molten aluminum with zinc.

Proposed Solution: Substitute ceramic blade or develop corrosion-resistant coating for the steel blade.

17. *Problem:* A-285 grade steel is unsatisfactory when used in facilities for processing molten zinc; the zinc penetrates through the steel.

Proposed Solution: Develop a ceramic coating for steel to prevent corrosion of the steel substrate.

18. *Problem:* The present refractory mix is inadequate in strength and thermal shock resistance for lining throats and channels of induction heating units for melting and heating zinc at temperatures of 750 C.

Proposed Solution: Develop improved refractory mix with improved strength and thermal shock resistance.

Lead:

19. *Problem:* There is no commercially available material which is resistant to molten PbO.

Proposed Solution: Develop a ceramic which is resistant to PbO and PbS. This would permit the conversion of PbS into Pb in one of several direct conversion processes.

Titanium, Hot Forming:

20. *Problem:* During hot forming of titanium at 705 C to 925 C any oxygen in the environment combines with and embrittles the titanium surface. This oxygen-rich surface must, subsequently, be pickled off. Coating material is applied to the titanium to retard oxidation. Materials presently used include T-50 graphite (with molybdenum disulfide) or a glass, 23-B.

Proposed Solution: Develop a glass with the following characteristics: (1) neutral or slightly reducing, (2) low viscosity at 705 C to 925 C and (3) good die lubrication properties.

21. *Problem:* Inconsistency of castable ceramic used to make hot forming dies by aircraft manufacturer. Unstable variable properties include gelling time, cold crushing strength and die surface hardness.

Proposed Solution: Either analyze the commercially available material and propose tighter quality controls necessary to standardize the product or develop new castable ceramic for this end item application.

22. *Problem:* Metal tooling for hot forming, brazing, and diffusion bonding is expensive to machine and frequently lacks dimensional stability under repeated cycling at elevated temperatures. Tooling is used for titanium and other high performance alloys at temperatures ranging from 540 C to 980 C. Present tooling materials include ferrous alloys such as Meehanite and H-11 steel, nickel base alloys, cobalt base materials, and fused silica ceramics (for gently contoured parts only).

Proposed Solution: Use cold-spray process for fabrication of fused silica to the problem of forming tooling of more complicated shapes. Also, try thixotropic cast fused silica to eliminate shrinkage on firing.

23. *Problem:* The lack of satisfactory structural (tensile) strength limits the use of ceramic hot forming tooling. Also, repairs or modifications are difficult or impossible. Another factor is the high degree of friction between the ceramic tooling and metal parts which prevents slippage of the metal during the forming process and is a contributing factor in the thermal expansion/contraction mismatch problem.

Proposed Solution: Same as previously discussed under #22. In addition, incorporate metal reinforcement for improved strength and consider fire polishing the mold surface to reduce friction.

24. *Problem:* Long-term need is for improved ceramic tooling material for hot forming titanium and other metals. Increased tool life, reduced tool fabrication costs and materials to withstand anticipated increases in forming temperature and pressure are required.

Proposed Solution: Initiate R&D project to address this and other tooling problems. (#22, 23, etc.)

25. *Problem:* Present ceramic materials used as tooling for hot forming titanium have a low production life expectancy due to stress risers during high pressure forming causing cracking, thermal cycling failure or failure due to handling. Fused silica castables, fused silica cements and foam block are the materials presently used.

Proposed Solution: Develop improved ceramic tooling with improved strength, toughness, and higher temperature capabilities. Forming temperatures of 955 C to 980 C are not exceeded as this is the limitation of fused silica castables and cements. Anticipated temperature requirements for titanium and nickel base alloys are 980 C to 1095 C and improved fused silica or other ceramics must be developed.

26. *Problem:* Metal press heated platens and tools for hot forming titanium have limited life due to warpage and deformation after usage at elevated temperatures and under high forming pressures. Inconel 802 is the material now used.

Proposed Solution: Same as others for ceramic tooling.

Molybdenum-Base Alloys:

27. *Problem:* Present ceramic investment casting molds are unsatisfactory for casting molybdenum-base alloys at temperatures approximately 1510 C as they melt or react with carbon and/or the alloying elements. The development of moly-base alloy cast turbine wheels or cast blade rings for automobile and truck turbines will depend upon skull melting and casting into investment molds.

Proposed Solution: Develop a ceramic which has no phase transformation, will withstand 1510 C and will not react with carbon or hafnium.

Refractory Metals:

28. *Problem:* Short life of extrusion dies for superalloys, refractory metals, and stainless steels.

Proposed Solution: Either improve on the process for facing die steels, H-11 or H-13, with ZrO_2 or HfO_2 or develop high temperature ceramic that is compatible with glass (as a lubricant) and temperatures of 1000 C to 1250 C.

29. *Problem:* Hard metals are now used as dies for hot extrusion of titanium and refractory alloys. They must be refurbished after a few runs; costly and excessive downtime of equipment.

Proposed Solution: Silicon nitride or silicon carbide may be suitable for this application.

Ceramic Cutting Tools:

30. *Problem:* Machining of high nickel alloys such as D979, 713, etc. Now done with carbide cutting tool inserts.
- Proposed Solution:* Develop improved ceramic cutting tools.
31. *Problem:* Ceramic tool bits used for machining cast iron are subject to chipping and breaking. Present tool bit materials are HP Al_2O_3 and Al_2O_3 -TiC composites.
- Proposed Solution:* Develop ceramic tool bits with improved impact resistance and fracture toughness.
32. *Problem:* Ceramic tool bits lack the rigidity and efficiency of carbide tool bits.
- Proposed Solution:* Same as #31.
33. *Problem:* The major shortcoming of ceramic tool bits is the brittleness of the ceramic. This tends to cause breakage when the tool bits are subjected to intermittent machining forces, interrupted cuts or vibration.
- Proposed Solution:* Same as #31.
34. *Problem:* Same as other problems associated with ceramic tool bits, plus the limitation on feed rates.
- Proposed Solution:* Same as others relating to ceramic tool bits.
35. *Problem:* Ceramic tool bits lack the strength and fracture resistance required for high speed machining of hardened steel rolling mill rolls. Tool bit materials currently used are hot-pressed alumina and alumina-titanium carbide cermets.
- Proposed Solution:* Same as previously discussed for ceramic tool bits.
36. *Problem:* Ceramic tool bits can be improved by upgrading the temperature capabilities and reducing the chipping. Materials now used include tool steels, carbides, and ceramics (alumina).
- Proposed Solution:* Same as previously discussed for ceramic tool bits.
37. *Problem:* Ceramic tool bits are limited to machining that does not transmit shock or vibration to the tool; no machining with interrupted or out-of-balance cut.
- Proposed Solution:* Same as others relating to ceramic tool bits.

38. *Problem:* Improved cutting tool materials are required to machine tungsten and carbide parts. Cubic boron nitride (CBN) is expensive, about \$100 per insert.

Proposed Solution: Develop new cermet with improved hardness and fracture toughness.

Pumps for Molten Metal:

39. *Problem:* Nonavailability of a high volume, portable pump for the transportation of molten aluminum. Pumps in various sizes are needed.

Proposed Solution: Develop refractory pump parts which do not react with molten aluminum at elevated temperatures and can withstand the environmental conditions.

40. *Problem:* Present pumps for pumping molten zinc are fragile and require excessive maintenance.

Proposed Solution: Develop refractory pump parts which do not react with molten zinc.

General Refractories:

41. *Problem:* Refractory brick are used to insulate the shell of a furnace used to sinter compacted tungsten powder billets in hydrogen atmosphere at temperatures up to 2300 C for four hours. The refractories fail by spalling and cracking due to thermal cycling of the furnace.

Proposed Solution: Develop high temperature refractory with low coefficient of expansion to improve thermal shock resistance.

42. *Problem:* In furnaces with temperature capabilities of 925 C it is customary to use high temperature metal linings.

Proposed Solution: To conserve strategic metals and improve heat insulation, substitute a ceramic felt or foam lightweight refractory.

43. *Problem:* Failure of steel chimney lining due to corrosion caused by the products of combustion.

Proposed Solution: Line chimneys with refractory that resists the corrosive fumes, gases and particulates (unless it is more economical to replace chimneys).

44. *Problem:* Conserve refractory metal materials.

Proposed Solution: Develop a ceramic refractory with structural strengths that withstands oxidation in hostile environment at 1650 C.

45. *Problem:* Improved refractories are needed for fusion casting of metals at temperatures of 1800 C to 2000 C.

Proposed Solution: Develop new refractories and new binder systems.

46. *Problem:* Refractories as crucibles and transfer tubes are subject to erosion during melting and spalling or erosion during the powder preparation of superalloys for high temperature gas turbine engine components. Ceramic refractories used as crucibles and transfer tubes are $\text{Al}_2\text{O}_3 + \text{ZrO}_2 + \text{SiO}_2$ crucibles and fused SiO_2 tubes.

Proposed Solution: Develop ceramic with improved erosion resistance under the stated environmental conditions.

OSHA:

47. *Problem:* Health hazard posed by asbestos material used as molten metal trough lining.

Proposed Solution: Substitution of mullite or other refractory fiber filler to replace asbestos. The lining must have mechanical stability at temperatures up to 1095 C.

48. *Problem:* Asbestos downspouts are used in aluminum reverberating furnaces.

Proposed Solution: Develop OSHA approved material for replacement.

49. *Problem:* Silica is the refractory used for lining the iron coreless furnace. There is no replacement refractory cement on the market.

Proposed Solution: Taking a long range viewpoint, OSHA may ban the use of silica (already in effect in Sweden) due to the danger of silicosis. A development program should be initiated to produce a low-cost substitute for silica refractory cement.

Miscellaneous:

50. *Problem:* The fastening of the ceramic dies is a problem in the hot die forging of metals.

Proposed Solution: Have the materials engineer and design engineer work together on this problem.

51. *Problem:* Porcelain enamel linings on aluminum are too soft.

Proposed Solution: Develop improved frits with increased hardness.

52. *Problem:* In the glass industry, metal rollers 100" long and 1-1/2" in diameter are used at 705 C. The rollers bend due to relaxation of residual stresses and nonuniform heating in the process.

Proposed Solution: Substitute a ceramic material which is stable at the operating temperature of the process.

53. *Problem:* When cemented tungsten carbide pieces are sintered in vacuum between 1300 C and 1600 C, they are placed on graphite plates coated with graphite dag or alumina slurry to prevent sticking. When weights are applied to prevent warpage the problem of sticking or contamination occurs.

Proposed Solution: Either improve existing graphite or coating materials that will not react or stick or develop a new ceramic that will not react with WC+Co or WC+Co+TaC+TiC at sintering temperatures.

54. *Problem:* High vacuum, reliable glass-to-metal seals are a prime requirement in many electronic components. The state-of-the-art is such that, on occasion, production problems are encountered.

Proposed Solution: Either develop improved specially formulated glasses or improve material quality control and processing technology.

55. *Problem:* The inability of metals and existing commercially available ceramics to withstand high temperature, thermal shock, and highly corrosive atmospheres.

Proposed Solution: Develop applications for silicon nitride in electronic parts such as resistance exothermal and magnetron-cathode; in mechanical parts such as abrasion-resistant nozzles, dies, and high-speed cutting tools; in aircraft parts such as jet engine components, brake linings, and rocket engine components.

56. *Problem:* High cost, long time and machinist expertise required for grinding refractories and dense industrial ceramics.

Proposed Solution: Use metal or resinoid-bonded diamond grinding wheels, if not already in use.

57. *Problem:* Fabrication of wing or control surface skins of graphite/epoxy composites require aerodynamically controlled tooling for lay-up and autoclave curing.

Proposed Solution: While outside the scope of this report, a possible solution to this problem might be the development of a lightweight castable ceramic foam with smooth, dense surfaces. It will probably have to be coated to prevent wetting and sticking during autoclaving.

58. *Problem:* A general problem exists when an attempt is made to substitute ceramics for metal as a component on existing equipment. The user or equipment manufacturer attempts to substitute on a one-for-one basis and is reluctant to make modifications to improve the performance of the ceramic.

Proposed Solution: The project must be conducted as a team effort with both material scientists and design engineers coordinating their input. For example, metal dies are cooled while ceramic dies should be kept hot. Fastening is difficult due to thermal expansion mismatch and both die configuration and method of alignment (for injection molding of metals) must be developed to prevent stress risers in ceramic dies.

59. *Problem:* The problem is the overall failure of ceramic materials in brittle fracture.

Proposed Solution: Development of a cermet-type material with improved ductility without sacrificing wear resistance and hardness.

60. *Problem:* Special steel alloys containing Ni-Mo-Mn are used for the crushing surfaces of impact-type crushers. Wear resistance is not satisfactory.

Proposed Solution: Development of a ceramic with high hardness and high impact strength.

61. *Problem:* In the industrial furnace market, delivery delays up to one year are common for cast 330 alloys used as conveyor parts such as rolls and radiant heat tubes, maximum strength limitations at 1205 C.

Proposed Solution: Provide ceramic substitute material with good thermal shock resistance and high impact strength and abrasion resistance.

62. *Problem:* Short life of "D" grade carbon pouring spout in phosphorus electric furnace due to erosion and chemical attack by calcium silicate slag; operating temperatures range from 1510 C to 1565 C. Several other ceramics tried without success.

Proposed Solution: Develop ceramic resistant to these environmental conditions. ZrB_2 :SiC:Graphite composite works, but is expensive.

63. *Problem:* Corrosive glasses high in borates and phosphates attack ceramic refractories at the melting temperatures of 1540 C and 1370 C, respectively. Platinum is now used for this application.

Proposed Solution: Conduct R&D studies to develop high temperature refractories which are chemically inert to these glasses.

64. *Problem:* A platinum-rhodium alloy is used for spinnerette bushings used to produce glass fibers. It is in short supply, is costly and expensive to reclaim and fabricate. The bushing must be able to withstand abrasion and erosion and the temperature of molten glass. The company has explored the use of ceramics, but without success to date.

Proposed Solution: Explore the use of improved, nonoxide ceramics for this application.

65. *Problem:* An inexpensive container for powder metal is needed for the hot isostatic pressing of aircraft parts. Steel is now used.

Proposed Solution: Dependent upon the temperature of compaction, develop glass which will soften at the specified temperature.

66. *Problem:* Heat checking of die casting shot sleeves and plungers used in the manufacture of aircraft engine components. Tool steel is used for the sleeves and plungers.

Proposed Solution: Develop ceramic sleeves and plungers for these applications.

67. *Problem:* High cost and possible material shortages of heat treat fixtures and racks. Nickel and chrome alloys are currently used.

Proposed Solution: Substitute ceramic fixtures and racks.

68. *Problem:* Stronger ceramic shells are required for fabrication of directionally solidified investment cast turbine blades. Shells must withstand 1565 C for one hour and not react with superalloys. Alumina is presently used.

Proposed Solution: R&D project to develop suitable ceramic.

Army Materials and Mechanics Research Center,
Watertown, Massachusetts 02172
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Ceramic materials
Refractory materials
Materials shortages

This report summarizes 171 replies to a questionnaire requesting information on ceramic material shortages and problems with ceramic refractories experienced by metal smelting and metalworking companies. Raw material shortages consist of seven ores; the main problems appear to be with imported materials. Problems range from depletion of the supply to sole source control of quantity. The problems relating to refractory usage within the metals industries are detailed according to industry and end-item application. Recommendations and possible solutions are included.

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