CERAMICS AND CERAMISTS ON THE MANHATTAN PROJECT: A NARRATIVE OF ACTIVITIES AT M.I.T. FROM 1944 TO 1946

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ABSTRACT

During World War II, a ceramic division of the M.I.T. Metallurgy Project (part of the Manhattan Project) was hurriedly organized to develop refractories for processing nuclear metals. In a brief space of less than two years, crucibles were developed from MgO, BeO, ThO₂, CeS, ThS, and some nitrides. Much of the success was due to the enthusiasm of several young engineers and scientists and some of their activities are described.

At the time of the Manhattan Project, commercially available crucibles for melting and refining the nuclear metals were not available of satisfactory purity. Requirements for impurities ranged in parts per million of some elements of high nuclear cross-section, e.g., boron, hafnuim, etc. Contrast these requirements with the composition of refractories manufactured in the forties — magnesia brick could contain as low as 83% MgO and refractories of "high purity" contained a minimum of 1 to 2% of impurities. Therefore to prepare suitable compositions and to maintain secrecy, refractories were studied at several sites, notably Chicago, Berkeley, Ames, and Los Alamos. It became evident that a large effort would be required.

Dr. John Chipman (of M.I.T. but temporarily at Chicago) had hired Wendell Keith for the Metallurgical Lab,

University of Chicago, from the University of Washington Ceramic Engineering Department at the end of the Spring quarter, 1943. His first job with Marion Thorsen, an Illinois ceramic engineering 1940 graduate who was in the Army and assigned to the project in 1943, was to move the ceramics work from under the University of Chicago stadium to the new Chemistry building in the Quadrangle during the summer of 1943. There work started with several additional associates -Dr. L.S. Foster, Dr. T.T. Magel, Dr. Morris Fine and They later moved to an old converted Nick Dallas. brewery which was designated Site B on the south side of the campus. In that facility two rooms were devoted to both office space and to the ceramic processing and fabrication effort primarily emphasizing small slip cast BeO and ThO, crucibles. Additional space was obtained in a high bay area just outside of the office/lab for a small furnace fired with two oxyacetylene welding torches. In addition, a graphite susceptor induction heated furnace was occasionally available in another lab which was found unsuitable for MgO because of reduction, resulting in smoke from reoxidation of the Mg fumes.

Gradually others joined the group. They included Lou R. McCreight, who had not yet graduated from Illinois and Bill Hillig, a Michigan mid-year chemistry graduate, who both joined in February 1944.

Plans to move and enlarge the group in both space and personnel were underway for Site B was inadequate. A meeting was held in Dr. Compton's office (President of the University of Chicago) attended by Wendell Keith, Dr. Ted Magel, Dr. Chipman, and consultants Dr. Louis Navias of the General Electric Co., and Karl Schwartzwalder and Dr. Helen Barlett of AC Spark Plug Co. Several ceramic engineering schools were suggested and Wendell was asked to visit them. Ohio State, Pennsylvania State, and Illinois were all found to be busy with other wartime projects. Iowa State was considered but because of indecision there, Dr. Compton announced, "Start moving to M.I.T. tomorrow".

A meeting of the group (Wendell, Marion, Bill and Lou) was immediately held (about noon on a Thursday in about the 3rd week of March, 1944). It was led by Dr. Chipman who told of the plan to move to MIT and how all would enjoy and benefit from it. (He was right). He

then asked, "Who can leave tomorrow?" Bill Hillig agreed to do so, and Lou agreed to go on Saturday. However Wendell said that he was going on Sunday in order to stop over in New York on Monday regarding some lab equipment.

At MIT there was already a small metallurgical project headed up by Al Kaufman which was joined for administrative purposes. The project was designated D.I.C. 6149.

The early days at MIT were spent cleaning out, packing and storing or shipping notebooks and lab equipment to Babcock and Wilcox (who had supported much of the MIT Ceramics work, pre-World War II).

About 20 lab rooms were eventually occupied scattered over several floors and sections of the main MIT building at 77 Massachusetts Avenue, Cambridge. The ceramics activities grew to about 60 people while the metallurgical groups grew to some 40 people for a total of about 100.

Top management was by Professors John Chipman, Morris Cohen and F.H. Norton. The latter two covered Metallurgical and Ceramic activities, respectively. The Metallurgical groups were doing a variety of R&D items related to uranium and beryllium, ranging from phase diagrams to precision casting. The latter activity, under Shad Marshall, later moved to Los Alamos where they did the fabrication of the actual fissionable parts for the first bombs.

In the Ceramics Division of the Metallurgy Project with Prof. F.H. Norton as head, several groups with separate objectives were organized. Transfers of people between groups occurred frequently and group leaders were changed several times so the definite compositions of the groups are difficult to recall. Since additional personnel were immediately required, the project requisitioned more ASTP (Army Student Training Program) engineering students from several universities. These young people, who had not yet finished their bachelors degrees, contributed much to the project success. They were Army personnel but, because of the project secrecy, wore civilian clothes. Later, in 1945, the Army decreed that all should wear regular uniforms. Imagine the other MIT peoples' surprise to see sixty

civilians going to and from several laboratories and the next day thirty civilians and thirty Army "noncoms" in uniform! The ASTP people could room where they pleased but had to keep their uniforms available to wear home on leave. In one case, in one rooming house an ASTP person was reported as a deserter!!

Other people were recruited for the project but the project secrecy created problems. Since draft boards could not be given any details, they sometimes questioned deferments. For example, Arnold Zais, a recent chemistry graduate on the project, was drafted. The Army read him the Acts at the camp on Cape Cod, issued him a uniform, and shipped him back to the project. Arnold had earlier been made a Group Leader, but was only a buck private in rank, yet was supervising several sergeants! It was suggested that he have his office door lettered: Arnold Zais, Private! Bill Hillig enlisted but the Army sent him back to M.I.T. in 30 days and with private's pay!

Four principal groups were created in the Ceramics Division: the production group, and groups on dense oxides, sulfides and nitrides.

THE CRUCIBLE PRODUCTION GROUP

MgO was used early on the project (and continued to be the principal crucible refractory for many years). However, early fabrication included BeO and ThO $_2$ but especially MgO. About 6,000 crucibles of MgO $_2$ were produced but far fewer of the other materials.

Highly calcined BeO was milled in a steel mill then acid washed both to remove the iron and to obtain a castable slip. A popular type of crucible was a conical shape having dimensions of about 3/4" diameter by about 1-1/4" high with 1/16" wall.

Occasionally, fused ThO $_2$ was crushed and milled then acid washed to make either a casting slip or a ramming mix (with organic binder) for preparing small crucibles (up to 1-2" diameter x 2-4" high).

By far the major effort was on MgO which was rammed by hand into crucibles at first. These crucibles were first fired in graphite molds and graphite lined induction furnaces to over 1700° C. using a big motor

generator in Prof. Nick Grant's laboratory. As MgO reduces under these conditions, a plume of magnesium vapor burned out of the top hole of the furnace. The hole gradually clogged from oxide deposition and had to be cleaned by prodding. When the hole choked, pressure would build up until the obstruction blew out. This caused a "fireworks" display as good as many Roman candles burning. Crucibles fired in this way had a glassy surface from evaporation of projecting particles.

As the project proceeded, Bob Fellinger and Hank Henderson designed an elegant apparatus for pneumatically ramming crucibles in a slowly rotating mold into which the mix was fed with a vibrating feeder. Norton describes this on page 133 in his 4th edition. After ramming the bottom was compacted in a hydraulic press and an elegant stripping device (also designed by Fellinger and Henderson) removed the crucibles safely from the mold. The combination of automatic ramming and stripping, with gas-firing, resulted in higher quality crucibles with lower rejections.

Firing was accomplished in several kilns. binder removal was usually done in an electric resistance furnace overnight. High temperature sintering was then done in high alumina brick-lined, gas fired kilns at temperatures usually in the range of 1700-1800°C. The primary "production" firing was done in two laboratory catenary arch kilns which were lined with fused 99% alumina brick. These , kilns are described by Norton (page 261, 3rd edition). One of the kilns was loaded with crucibles afternoon, including Sunday but not Saturday, preheated overnight to a red heat with simple atmospheric gas-air burners made of standard pipe fittings and then finished off to 1700°C or so with a large Maxon premix burner the following morning. That kiln would be allowed to cool overnight, then be unloaded at a temperature of a few hundred degrees in the morning and reloaded in the afternoon when the furnace was still pretty warm. Unloading was done with tongs while loading had to be done by hand due to the relative fragility of the ware. The kilns were often warm enough to cause Lou's hair oil to smoke during the loading operation!

These kilns had setting spaces for about 40-50 of three inch diameter or 30-36 of four inch diameter MgO crucibles. Sometimes by using a special MgO setter a small crucible would be set inside of a large crucible. Both were set upside down to help keep them free of internal contamination.

The raw material used for these crucibles was fused magnesia. The specification called for less than 100 ppm B, a common impurity in MgO. This specification was obtained by mixing equal parts of a fused "CP" magnesia containing approx. 200 ppm B with fused Indian magnesite containing approx. 6 ppm B but approx. 4% SiO₂. Fortunately, this latter material was available because low B content was necessary in fused magnesia used for sheathed electrical heaters.

Several acted as Group Leaders of this production group during the 18 months of operation including Wendell Keith, Ted Magel, Leroy Moody, John Ohlson and Arnold Zais. Other members of the group included Lou McCreight, Marion Thorsen, Larry Garvey, Todd Ross, Nick Dallas, Fay Cunningham, Suzanne Lipsett, and others.

THE DENSE OXIDE GROUP

As impervious crucibles might be required, an oxide group was organized by Dr. Helen Barlett, who was on loan from the A.C. Spark Plug Div. of General Motors. Members of the group included O.J. Whittemore, Betty Livingstone and Virginia Mahady.

Their first objective was to develop impervious MgO crucibles of the same 2% SiO $_2$, <100 ppm B composition described above. To achieve fine enough particle size, fused MgO was ball-milled in a non-aqueous liquid, chosen as 95% ethyl alcohol. This milling medium had many advantages, including improving morale at the 1944 Christmas party.

After evaporating the alcohol, the fine MgO required a binder-lubricant for pressing. Barlett introduced phenol formaldehyde resin which was then being used as a thermo-setting binder-lubricant by AC but many problems resulted with the crude mold heating apparatus hastily assembled. Whittemore suggested Carbowax 4000, a water-soluble polyethylene glycol wax, and this

proved successful in slightly warmed molds. Bartlett had shown that large amounts of binder-lubricants could be used (up to the pore volume of the compact) and 12% wax was used. Crucibles were pressed at about 210 MPa. The wax was very carefully burned out by heating overnight to 500° C, holding the temperature at 150° C for one or two hours. The crucibles were then fired at 1700° C in an oxy-propane kiln. Several hundred impervious MgO crucibles were produced ranging from 0.5 to 4 cm diameter.

When the procedures for producing impervious MgO crucibles were worked out, the group began work on producing dense BeO crucibles. The raw material used was a light fluffy oxide of 99.8% BeO supplied by Foote Mineral Co. which had been calcined quite variably. Re-calcining was required and many problems occurred. About this time, Dr. Barlett was called back to A.C. and Whittemore took over the group. Others joining the group were W.W. Galbreath, Jr., J.E. Roman, Jr. and O.L. Widmoyer. Calcining at 1300°C for 3 hours was found optimum for preparing impervious crucibles, while 1400°C calcines gave porosities of from 20 to 25%'. Particle size distributions by sedimentation (the hydrometer method also described on pages 542-547 in ref. 3) gave some indications of a desirable distribution but were not definitive. Some of the first electron microscope photos later showed the grain growth of beryllia during calcining and that aggregates were causing confusion . The calcines were wet ballmilled 10 hours. A dispersing agent (Daxad 23, Dewey and Almy Chem. Co.) was used for the particle size determinations so it also was added to the mill charge resulting in better reduction of aggregates. milled batch was dried and mixed with 14% Carbowax (in a water solution), granulated through 14-mesh and dried. Crucibles were pressed at 210 MPa in tool-steel tapered molds. Sticking to the die occurred frequently until the Daxad 23 was added (only in the amount of 0.1%). The crucibles were then fired similarly as those of MgO but to 1800°C and some weight loss occurred during firing (probably due to reaction with water in the kiln gases). Norton later described this work (pages 307-312, ref. 3). Several hundred impervious crucibles were produced, ranging from 0.5 to 4 cm diameter.

Bill Galbreath took on the problem of making impervious ThO crucibles. Calcined thoria was quite variable and sedimentation curves showed the unsatisfactory material had a mean size of 22 $\mu\,\text{m}$ where the satisfactory material was 8 μm . Dense crucibles could not be produced from pure thoria so a number of additives were tried and 2% ZrO found best. Processing was similar to the MgO and BeO crucibles: milling 10 hours, adding 6% Carbowax, pressing at 210 MPa and firing at 1800°C. The crucibles were nonporous, with densities from 9.55 to 9.70.

Large porous beryllia crucibles which had earlier been produced by the Production Group were later produced by Bill Galbreath for the use of the metallurgists studying beryllium. These were from 10 to 25 cm in diameter and up to 40 cm high. These were hand-rammed from a mixture of coarse beryllia and fired to 1700 C in a catenary kiln similar to that used for large porous magnesia. By varying processing, lifes were extended from one to about four heats in melting beryllium. Used crucibles were crushed, recalcined and used in later batches.

At the time of the project, only acid salts and metal fumes of beryllium were considered toxic. Later, fine (especially calcined) beryllia was found to be selectively toxic. Several people working on the project came down with "berylliosis", a serious lung condition, some with delayed cases several years after working on the project, and several died later.

THE SULFIDE GROUP

Eastman and Brewer 11 had developed preparation procedures for several sulfides which thermodynamically looked promising as refractory crucibles. A group was organized, first under A.L. Johnson, later I. Amdur to produce the most likely sulfide -- CeS. Cerium oxide was placed in graphite containers through which $\rm H_2S$ gas was passed while heating the containers at $1100~\rm C$, resulting in $\rm Ce_2S_3$. This product was mixed with cerium hydride and reacted in vacuum at about $2000~\rm C$ to obtain CeS. The CeS was steel ball milled with ethyl ether and cetyl alcohol, then rammed and pressed at 140 MPa in hardened steel molds. The binders were removed at

 $125^{\circ}\mathrm{C}$ in vacuum, the crucibles sintered resulting in porosities from 1 to 10%. One or two hundred were produced, up to 8 cm diameter.

In addition, several ThS crucibles were prepared in a similar way. Norton also mentions these materials (pages 330 and 331, ref. 3).

THE NITRIDE GROUP

A group was organized under L.S. Foster to study nitrides and included Lloyd Forbes, Joe Tvrzicki, Larry Friar, and Bill Smith. A few small crucibles of TiN were produced but the work was discontinued due to pressures of preparing the magnesia and sulfide crucibles.

SUPPORT

A number of other people on the project contributed to the success. Bill Hillig determined particle size distributions on all of the materials mentioned (except MgO). He was also assisted by Alice Carney who also determined thermal expansion coefficients to 1400°C of several of the products by the telemicroscope method.

Bert Ball organized an efficient procurement group (sometimes called Ball's harem because it included several attractive girls). This group procured materials rapidly and supervised shipping and many other details.

Ed Read set-up a chemical analysis laboratory, performing quantitative analyses on the large number of unusual compounds.

SECURITY

When Lou joined the project in Chicago, he was told the code names for the sites at which the work was being performed as well as the code names for the materials and in some detail of how to build atomic bombs. The point that two pieces of Uranium 235 must be rapidly brought together was emphasized, however the then suggested mechanism was to be by ground impact which, of course, was unsuitable for several reasons.

An interesting observation was that the code name for BeO was BaO which was obtained in drums from the Brush Beryllium Co.! Uranium was coded as tuballoy and thorium as myrnalloy (a play on that actress' name)!

In Chicago, physical security was ostensibly quite tight, however when moved to MIT a more casual approach to security seemed to work well. For example, as previously mentioned, the lab rooms were scattered about the Institute so there was no central guarded enclave. Near the end of the war, when the Atomic Bomb was announced, most MIT'ers as well as Bostonians thought that the atomic bomb work at MIT was being done in the Radiation Lab which was actually devoted to radar R&D. In general, the MIT project people were not told that they were working on an atomic bomb and those from Chicago were told not to pass that information on to them.

The primary ceramics lab at MIT contained the kilns and in spite of a big ventilating hood, was uncomfortably warm. The windows in the hallway doors were therefore replaced with heavy mesh and plywood internal panels. Between that feature and the fact that the room was a semi-basement room with windows and a driveway/hallway running along the outside wall, made for very casual security.

After about a year of operation, about 3/4 of a ton of scrap crucibles had accumulated. It was thought that anyone having an idea of what the project was working toward could get a good idea of the progress by seeing and analyzing these scraps. So instead of dumping them in a trash collection area just down the hall from the main labs, it was first planned to take them to a landfill operation where they would be pushed into the ocean, then it was decided to haul them out into the North Atlantic Ocean for disposal. There was a convenient way to do this aboard a Navy ocean-going tug which was being broken in by using it as an Army freight and supply ship to supply POW's located on some of the islands in Boston Harbor. So one Saturday morning about March of 1944, several project personnel, accompanied by an FBI man as a guard, went out to do this job. It turned out to be a long day with no lunch, only some thick, strong coffee for sustenance

and with a cold raw wind, especially out on the Atlantic. The FBI man was upset with people for skimming some flat disc-shaped crucible covers over the waves! The project people were also impressed with the quality and quantity of food delivered to the POW's every day. There were cases of lettuce and vegetables plus chickens.

CONSULTANTS

Several well-known, senior chemists and ceramists served as consultants. These included Dr. Louis Navias of the G.E. Research Labs, Dr. Frank Spedding of Iowa State University (whose project developed the uranium reduction process), Vic Duplin of Babcock and Wilcox, and Karl Schwartzwalder of A.C. Spark Plug.

Once a dinner was held for the consultants in a private dining room at the Parker House. There were 4 tables of 8 each composed of the Consultants and selected MIT staff and management personnel. At one table they started telling jokes after dinner. One in particular elicited so much laughter that Prof. Chipman requested that it be repeated for all to hear. Manny Gordon was prevailed on to do the honors, which he did very well, and this set off a fun-filled evening of visiting and relaxing from the concentrated hard work on the project.

IN RETROSPECT

The project accomplished many objectives in the relatively short time of about 18 months. During the project, many thought it a "boon doggle". However, many worked very hard and productively to produce the crucibles. Lou recalls working until 1 AM and deciding to just sleep on some lab coats on a lab bench rather than to walk across the Mass. Ave. bridge to his room. In the early days of the project some were on an hourly pay grade, which with overtime added up to quite a good income. Eventually they were "promoted" to salary status (\$200/month) and later given a \$5/month raise! both of which represented a marked decrease in their income.

Many humorous events occurred. Unsatisfied with what was considered inefficiency, an order came down to the

Production Group to send up daily individual activity reports. With much glee, each member wrote several pages each day, including time for conversations regarding work, trips to the bathroom, etc. After several days of stacks of reports, an order came down to discontinue activity reports!

The Christmas '44 party was arranged in the Chemistry Department's faculty lounge which was reserved for a "Business Meeting" late one afternoon a few days before Christmas and a case of 250 ml beakers was checked out to use for glasses. A colorful punch from ginger ale and green sherbert was suitably diluted with "ball mill" alcohol. When the punch ran out they quickly bought a case of cokes which were emptied into the punch bowl and again diluted with ice and ball milling fluid! The party continued for many over at Barb Barnett's apartment. At the start of the party, a photo was taken of those present (Fig. 1).

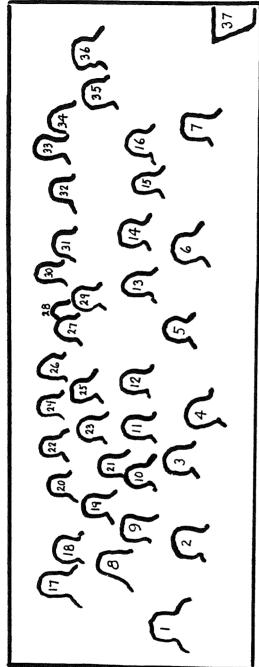
On August 7, the bomb was used in the war with Japan, ending the war and saving many American lives, which would have included many friends of the project personnel. But the MIT project was small compared to the large sites and we were required not to even state we were on the project. This ended soon although technical details were kept secret for many years.

After the war, there was a large exodus from the project. Lou McCreight returned to Illinois for undergraduate and graduate study. Wendell Keith and O.J. Whittemore went to the Norton Co. Wendell arranged the manufacture of MgO crucibles there and Whittemore moved the crucible production there. Bill Hillig ended up in the General Electric Research Laboratory, Ted Magel with Allegheny-Ludlum, Bob Fellinger with Mechanical Engineering at Iowa State, Roy Moody with General Electric; Bill Galbreath with Babcock and Wilcox, Bill Smith with SuperTemp, etc. etc.

ACKNOWLEDGMENT

We wish to thank Wendell Keith for his suggestions. Also we apologize to those we missed -- 44 years is a long time!





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1.	Bert Ball	16.	Ed Vivian	34.	Barb Heyer
5.	Al Kaufman	17.	O.J. Whittemore	35.	Lou McCreight
4.	Lloyd Forbes	19.	Hank Henderson	36.	Bob Fellinger
ς.	Suzanne Lipsett	22.	Alic Carney	37.	250 ml beakers!
∞	Paul Gordon	24.	Dorothy Baker		
10.	. Manny Gordon	25.	Fay Cunningham		
11.	. Barb Barnett	26.	Betty Livingstone		
12.	. F.H. Norton	28.	Virginia Mahady		
13.	13. Wendell Keith	31.	Sarah Hammer		
14.	14. Ted Magel	32.	John Chipman		
F					

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