Report of Investigations 8443

Shape-Casting Titanium in Olivine, Garnet, Chromite, and Zircon Rammed and Shell Molds

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SHAPE-CASTING TITANIUM IN OLIVINE, GARNET, CHROMITE, AND ZIRCON RAMMED AND SHELL MOLDS

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ERRATA

On page 12, the last line of paragraph 1 should read as follows:

and 145 psi when No. 180 olivine was employed.

SHAPE-CASTING TITANIUM IN OLIVINE, GARNET, CHROMITE, AND ZIRCON RAMMED AND SHELL MOLDS

by

R. K. Koch[1] and J. M. Burrus[2]

ABSTRACT

In seeking substitutes for such critical metals as chromium, cobalt, and nickel, the Bureau of Mines investigated techniques for shape-casting titanium in rammed sand molds. Castings were made in olivine, garnet, chromite, and, for comparison, zircon. It was found that commercial-grade castings up to 31 lb (maximum capacity of furnace) could be made in zircon or olivine molds if a zirconia mold wash was used. Castings up to 4 lb could be made in chromite molds, but heavier castings suffered from some mold-metal reaction. Garnet molds were found to be unsatisfactory for castings of all sizes because of gas blows and rough surfaces. In other tests shell castings of acceptable quality were produced in waterglass-bonded zircon and olivine molds up to weights of 8 lb, but chromite molds were unsatisfactory because rough casting surfaces were caused by mold-metal reaction. Unlike the currently used commercial processes, neither the rammed-sand nor the shell-molding processes developed by the Bureau of Mines generate noxious fumes at any step.

INTRODUCTION

Among the long-range goals of the Bureau of Mines are (1) minimizing requirements for mineral commodities through conservation and substitution, (2) minimizing environmental conflicts such as air pollution, and (3) maximizing productivity while reducing capital, labor, and energy requirements in the mining and minerals processing industries. Progress toward all three of these goals is sought in this research, which is aimed specifically at determining the technical feasibility of producing titanium castings in low-cost sand molds by low-energy, pollution-free processes.

Substitution of titanium for stainless steel and superalloys in castings would enable conservation of our limited resources of nickel while

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reducing our dependence on imported chromium. In the transportation industry, because of titanium's high strength-to-weight ratio (titanium is only 56 pct the weight of steel), an energy savings could be realized if the cost of commercial-grade titanium castings can be reduced. Substitution of lighter weight titanium castings for heavier steel or brass castings would result in sizable fuel savings in marine and land carriers. Rudinger and Ismer (15)[3] report that the substitution of titanium for steel in connecting rods in various engines resulted in power output and efficiency increases of 17 to 27 pct (theoretical maximum of 43 pct).

To achieve these desirable ends, however, it will be necessary to reduce the difficulties in, hence lower the cost of, producing commercial-grade titanium castings. The currently used rammed graphite process, which was initiated separately by the Bureau of Mines (8) and by Field (12) in 1956, consists of ramming an aqueous mixture of graphite powder, cornstarch, petroleum pitch, and dextrin around a reusable wood or metal pattern. This process, largely developed through additional Bureau of Mines research (4-5), still is beset by problems, among which is that large volumes of noxious gases are generated at one or more steps. These problems include:

1. The flowability of graphite molding mixes is so poor that molding machines can only be used on very shallow draw patterns.

2. The green strength is so low that the molds must be cured before assembly, and large cores tend to slump before cure strength is developed.

3. The curing process consists of a 24- to 48-hour air set followed by 4 to 5 hours at 425° F. Then the molds are fired in a reducing atmosphere for 6 to 12 hours at 1,600° F and furnace cooled; the firing cycle usually requires 24 hours. The long turnaround time on curing and firing requires considerable floor space and equipment.

4. Molds frequently warp and/or crack on firing, which makes dimensional control extremely difficult.

5. During the firing cycle large volumes of organic gases are evolved from molds and cores; consequently, the furnace offgases are heavily laden with a great variety of partly oxidized organic compounds.

6. Castings must be poured centrifugally to counteract the effects of mold gases generated by reaction of residual binder and molten titanium.

7. A very high percentage of the castings require weld repair because of gas porosity.

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[3] Underlined numbers in parentheses refer to items in the list of references preceding the appendixes.

8. One-fourth of the expended molding mix is discarded each cycle to reduce the impurity accumulation from expended binders.[4]

9. Castings of greater than 6- by 6-inch cross section frequently encounter carbide formation (scabs) on the casting surfaces. Even in molds of smaller cross section, titanium carbide scabs are formed if the molds are rammed too softly (if the molds are rammed too hard, they warp or crack on firing). The titanium carbide scabs are too hard to be removed by machining or grinding, so the affected castings are usually scrapped.

10. Core knockout represents a cost problem because of the hand labor involved. Rammed graphite cores become so hard during pouring that they have to be removed with chipping hammers. Considerable time and effort is expended on the removal of complex cores.

11. All castings of less than 100 lb must be cast centrifugally, which entails more labor for mounting the molds and balancing the load for spinning than is encountered in static casting.

Consideration of these problems led the Bureau of Mines to investigate alternate processes, seeking a lower cost, improved rammed molding process for titanium casting that would also eliminate or minimize production of noxious gases. Its first study was on zircon sand bonded by waterglass (14). The results of that investigation showed that commercial-grade castings up to 16 lb could be made in zircon molds, and that most of the problems associated with rammed graphite molding were overcome. Unfortunately, during the course of the investigation the cost of zircon foundry sand tripled, so much of the cost benefits were lost.

Therefore, this investigation was undertaken to compare lower cost foundry sands with zircon for casting titanium in rammed molds. And the lowest cost sand studied, olivine, was found to compare very well with zircon. In addition, the methods tested do not generate noxious fumes at any stage of the molding or casting process.

A second intent of this investigation was to determine the technical feasibility of producing titanium castings in waterglass-bonded shell molds. Reusable-pattern shell molding is often the lowest cost process for the production of ferrous and nonferrous castings that range in weight from a few ounces to about 400 lb. Shell molding produces castings with smoother surfaces, better detail, and greater dimensional accuracy than can be obtained by rammed-sand molding. However, shell-mold patterns are considerably more expensive than rammed-sand patterns. Therefore, the number of castings to be made (amortization of pattern costs), the surface smoothness and detail required, the casting weight, and the amount of machining required must all be considered in determining which process (rammed or shell) is the more economical for casting a given shape.

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[4] One foundry is now recycling 98 pct of the expanded molding mix with a 12 pct addition of new binder.

At the time this investigation was started, no titanium founders were making castings in reusable pattern shell molds. However, while the research was in progress, one titanium founder began casting in organic-bonded zircon shell core molds. The proprietary process used by that founder produces excellent castings that appear to be superior to rammed graphite castings with regard to detail and integrity (freedom from gas porosity).

Shell core molding machines differ somewhat from shell molding machines, but a sand mix that will work in a shell core machine will work in nearly all shell molding machines.

The preliminary investigation on waterglass-bonded shell molds, reported here, differs from the proprietary process in that no organic binders are employed. As a consequence, no petrochemicals are consumed, and no poisonous gases are generated during any step of the molding or casting process.

MATERIALS

All materials used in this study except Mouat chromite were readily available from foundry supply houses. The Mouat chromite used was material remaining from smelting studies conducted by Hunter and Banning, who described the characteristics of the Mouat deposit in Montana for the Bureau of Mines (13). This Mouat chromite is submetallurgical in grade; it was mined from the Montana deposit for stockpiling by the Defense Materials Procurement Agency between 1953 and 1961.

Purchased chromite originating in South Africa was also used in this study at a cost of $175 per ton in 100-lb sacks. In text it will be referred to as either "chromite" or "purchased chromite" to distinguish it from the "Mouat chromite."

The zircon sand used was from Australia because domestic zircon was not available from local foundry supply houses. The cost was $342 per ton in 100 lb sacks.

The olivine sand was mined and crushed at Mt. Vernon, Wash. Olivine No. 70, No. 90, and No. 180 sands were used during the course of this study. The cost was $67 per ton in 100-lb sacks.

The garnet sand was mined and processed near Fernwood, Idaho. Two sizes were used, M-80 and M-100. The cost was $129 per ton in 100-lb sacks.

The chemical analyses for all of the sands are given in table 1, and size distributions are listed in table 2.

TABLE 1. - Chemical analyses of as-received chromite, zircon, olivine, and garnet sands, pct

Sand	Cr_2O_3	FeO	MgO	SiO_2	Al_2O_3	CaO	ZrO_2	L.O.I.[1]
Mouat chromite....	42.3	24.4	12.1	3.8	16.7	1.4	(2)	0.62
Chromite..........	47.5	25.2	10.2	.7	12.3	.04	(2)	.39
Zircon............	(2)	.1	(2)	34.5	.1	.10	64.9	.15
Olivine No. 70....	(2)	8.30	46.1	42.5	.45	.41	(2)	.31
Olivine No. 90....	(2)	8.48	42.0	43.7	.65	.54	(2)	.30
Olivine No. 180...	(2)	8.65	45.0	41.4	.60	.49	(2)	.11
Garnet M 80.......	(2)	32.0	2.4	37.5	18.4	1.15	(2)	.46
Garnet M 100......	(2)	31.8	2.5	37.3	18.1	1.09	(2)	.51

[1] Weight loss after 1/2 hr at 1,470° F (900° C).

[2] Less than 0.05 pct.

TABLE 2. - Weight distribution of as-received chromite, zircon, olivine, and garnet sands

USA standard screen size	Weight retained, pct							
	Mouat chromite	55/60 chromite	Zircon	Olivine No. 70	Olivine No. 90	Olivine No. 180	Garnet M-80	Garnet M-100
Minus 20 plus 50 mesh......	38.2	54.1	0.8	27.6	(1)	(1)	50.6	(1)
Minus 50 plus 70 mesh......	17.8	21.6	.4	39.4	15.4	0.1	28.9	(1)
Minus 70 plus 100 mesh.....	14.6	15.4	39.9	22.5	34.4	.5	18.0	10.2
Minus 100 plus 140 mesh.....	9.6	6.1	46.7	5.3	22.7	5.4	2.3	82.2
Minus 140 plus 200 mesh.....	9.3	2.2	10.0	3.2	16.8	49.3	.1	7.4
Minus 200 plus 270 mesh.....	4.9	.4	1.9	1.1	7.1	27.4	.1	.1
Minus 270 mesh.........	5.6	.2	.3	.9	3.6	17.3	(1)	.1
AFSgfn[2]	83	54	94	61	103	182	50	100

[1] Less than 0.05 pct retained.

[2] American Foundrymen's Society grain finess No.

All of the waterglass employed in this study was grade D. In common with the many grades of waterglass, grade D is an aqueous solution of Na_2SiO_3 (sodium metasilicate) and SiO_2 (silica). Grade D has a silica to sodium oxide (Na_2O) ratio of 1.97:1, which is lower than the SiO_2:Na_2O ratio of most commercial grades of waterglass. The grade D solution has a viscosity of 50.5 Baume, and specific gravity of 1.53, and contains, in pct, 29.0 SiO_2, 14.7 Na_2O, and 55.9 H_2O.

The binding action of waterglass is due to the release of colloidal silica. The simplified reactions taking place can be represented as:

$$Na_2O \cdot nSiO_2 + CO_2(g) + H_2O(l) = Na_2CO_3 + nSiO_2 + H_2O(l) \tag{1}$$

and

$$Na_2O \cdot nSiO_2 + H_2O(l) = Na_2O + nSiO_2 + H_2O(g) \tag{2}$$

where n varies depending on the grade of waterglass employed.

Reaction 1 forms the basis of CO_2 molding. The reaction is rapid at 65° F. However, it was found that the sodium carbonate (Na_2CO_3) produced in reaction 1 caused gas blows in titanium castings. Therefore, reaction 2 was employed in this study, although it is slow and requires heat for water removal in order to develop strength.

MELTING AND POURING

Titanium was melted and poured by two different methods. One method was the consumable-electrode vacuum arc melting furnace (skull-casting furnace). Larger models of this furnace are used by all titanium founders for melting and pouring. The skull-casting furnace, which has been thoroughly described by Beall and others (7), will not be detailed here.

Two different crucibles were used in the skull-casting furnace. The smaller crucible was capable of delivering 16 lb of titanium to the mold. This crucible was used for all of the pours except the 6-inch cubes. The cubes required a larger crucible, which was capable of delivering about 31 lb of titanium to the molds.

The second method for melting and pouring titanium was the patented (6, 9) inductoslag furnace described by Clites and Beall (10-11). This furnace also can accomodate crucibles of various sizes. The crucible used for this investigation was able to deliver 8 lb of titanium to the mold. The furnace atmosphere and chamber pressure were controlled by a vacuum pumpdown, and then backfilling with helium to 4.75 psia.

RAMMED SAND CASTING

Patterns

Six patterns were used in the rammed sand casting investigation. These patterns were a cored 1-inch pipe tee (nominal 1/8-inch wall), a 12-inch tapered wedge, a 1/4-inch test bar gang, a bell with a nominal 1/8-inch wall, a 4-inch solid cube, and a 6-inch solid cube. The tee, wedge, test bar, and bell patterns are illustrated in figures 1 and 2.

Zircon Rammed Molds

These molds were prepared from the foundry-grade zircon sand listed in tables 1 and 2. One hundred parts by weight (pbw) of dry sand was

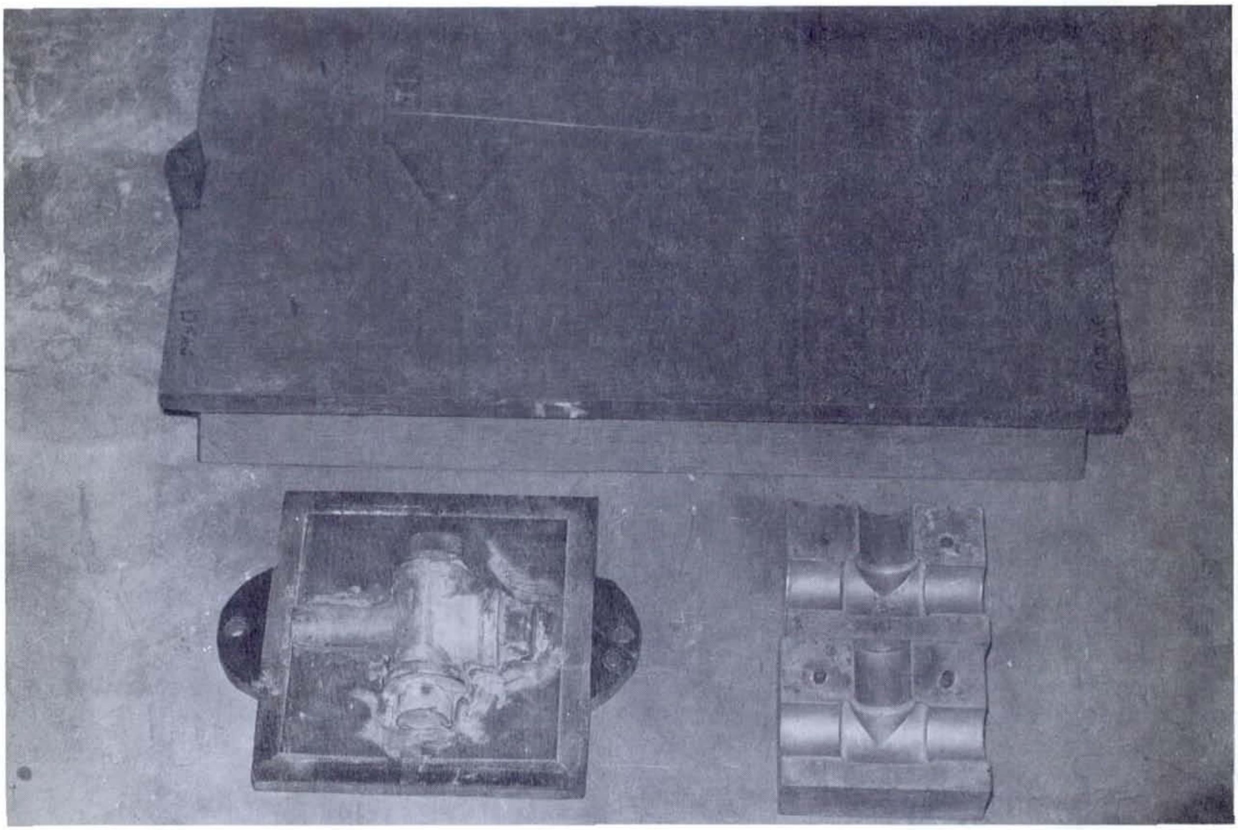

FIGURE 1. - Board mounted 12-inch wedge pattern, and 1-inch pipe tee pattern with core box.

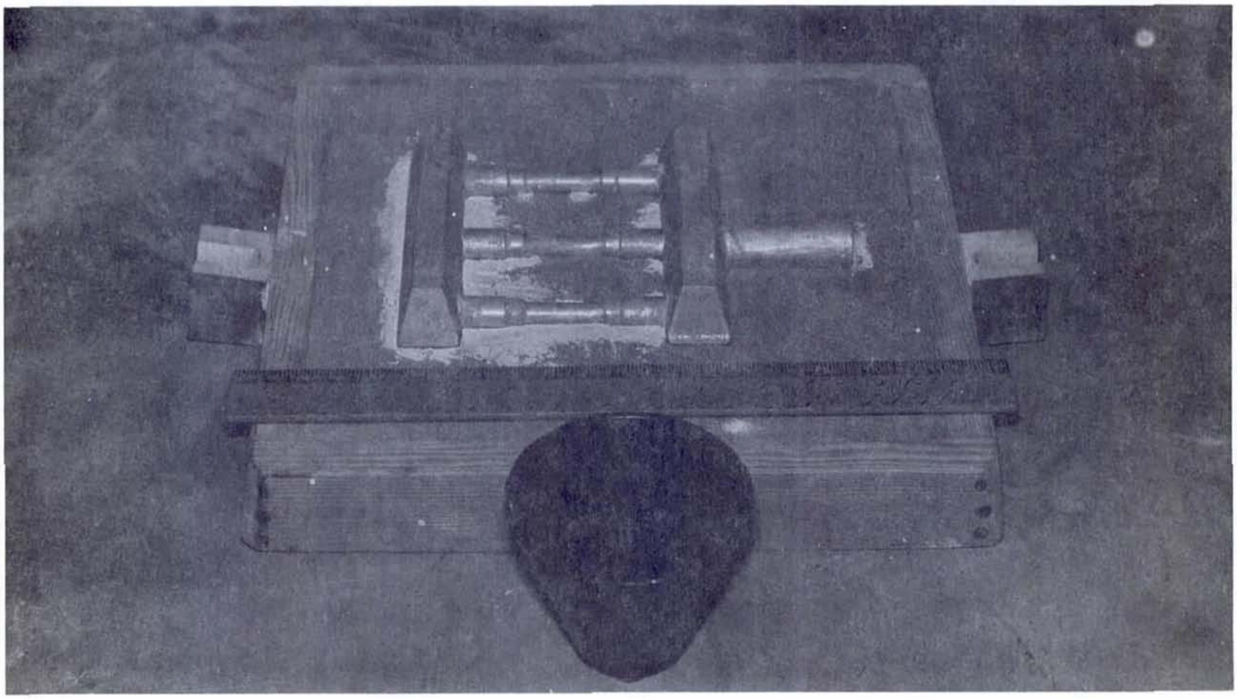

FIGURE 2. - Test bar pattern with followboard, and loose bell pattern.

mixed in a rotary cement mixer for 10 minutes with 3.8 pbw waterglass dissolved in 2.5 pbw water.

The mixed sand had a bench life (working life) of about 2 hours at 65° F and a relative humidity of 60 pct. That is, after 2 hours exposure to circulating air, heap sand began to crust on the surface which adversely affected the molding properties of the sand. The overaged rammed sand tended to stick to the pattern, and the molds tended to crack during curing. The working life could be extended to 3 or 4 weeks if the mix was stored in a closed container.

Specimens of this mix had average tensile strengths of 608 psi after curing at 480° F. The tensile specimens were compacted with three blows from a standard 14-lb rammer in a standard briquet mold and pulled on a universal testing machine in accordance with ASTM designation C 190-72 (2). Details of specimen preparation and testing are given in appendix A. Dry permeability of the sand mix was determined in accordance with the procedure given in the AFS Molding and Core Test Handbook (1). The average dry permeability number was 44±1 for nine samples.

Because the green strength of the molding mix was very low (too low to be measured with available equipment), copes and drags were not assembled until after the mold halves were cured at 480° F for 1 hour per inch of mold thickness. Cores were cured the same way. A few molds and cores were fired at 1,600° F for comparative purposes.

Detailed descriptions of the results obtained for the bell, wedge, and 4-inch cube castings are given in a previous publication (14) and will be summed up here as being satisfactory in all respects as commercial-grade castings. The test bar and 6-inch cube patterns were not available when the earlier report was written, so they will be mentioned in some detail here.

Four test-bar molds were prepared from the sand mix described previously. The molds were poured two at a time in the skull casting furnace. Two of the molds (heat No. 1, table 3) were cured at 480° F, and the other two (heat No. 2, table 3) were fired at 1,600° F. None of the molds had protective washes. The mechanical and chemical results are shown in tables 3 and 4, as well as the applicable requirements American Society for Testing and Materials (ASTM) per standard B 367-69 (3).

TABLE 3. - Mechanical data for titanium skull castings in zircon, olivine, and chromite rammed molds

Heat No.	Test bar No.	Ultimate tensile strength, ksi	Yield strength (0.2 pct offset), ksi	Elongation (in 1 inch), pct	Reduction in area, pct
			ZIRCON		
1.................	1	63.8	52.6	34	59
1.................	2	63.0	52.4	32	64
1.................	3	63.0	52.2	37	66
1.................	4	62.9	51.7	36	63
2.................	1	56.8	47.2	41	71
2.................	2	62.8	46.5	44	67
2.................	3	55.8	45.5	43	70
2.................	4	56.1	46.7	36	70
2.................	5	56.0	46.1	46	72
Avg.............	-	60.0	49.0	39	67
			OLIVINE		
3.................	1	72.0	60.4	27	44
3.................	2	71.8	59.5	28	44
4.................	1	57.9	45.3	38	69
4.................	2	58.3	44.9	36	68
4.................	3	58.2	44.8	37	68
4.................	4	60.2	47.8	36	67
4.................	5	60.0	46.1	38	67
Avg.............	-	62.6	49.8	34	61
			MOUAT CHROMITE		
5.................	1	61.1	50.7	36	61
5.................	2	61.9	51.8	36	62
5.................	3	62.7	50.5	36	55
Avg.............	-	61.9	51.0	36	59
			ASTM GRADE[1] C-1 (MINIMUM)		
-	-	35.0	25.0	24	(2)
			ASTM GRADE[1] C-3 (MINIMUM)		
-	-	65.0	55.0	15	(2)

[1] Minimum permissible values per ASTM B 367-69 (3).

[2] No specified minimum value.

TABLE 4. - Chemical analyses of test bars listed in table 3

Heat No.[1]	Test bars[1]	Concentration, ppm				
		O	H	N	Cu	C
ZIRCON						
1	1	1,060	38	146	100	60
1	2,3,4	1,140	37	125	100	80
2	1,2	880	40	10	100	90
2	3,4,5	945	41	30	100	120
OLIVINE						
3	1,2	1,470	62	165	500	140
4	1,2,3	940	72	57	500	120
4	4,5	1,040	62	54	500	120
CHROMITE						
5	1	980	44	133	100	70
5	2,3	1,060	45	142	200	90
ASTM GRADE C-1[2]						
-	-	1,800	100	300	1,000	1,000
ASTM GRADE C-3[2]						
-	-	3,500	100	500	1,000	1,000

[1] Heat and test bar numbers refer to table 3. Test bars from the same mold were composited for analysis.

[2] Maximum permissible concentrations per ASTM B 367-69 (3).

It can be noted in table 3 that only four of the six bars cast in zircon heat No. 1 and only five of the six cast in zircon heat No. 2 were pulled. The reason for this is that minute flaws were detected when the gage lengths of the test bars were machined down from the ascast 3/8-inch diameter to the 1/4-inch test diameter. In addition to the test bars, a 6-inch cube was cast in a zirconia-washed, rammed zircon mold to determine whether the increased time at elevated temperature at the mold-metal interface would substantially increase the extent of mold-metal reaction. The casting produced is shown in figure 3. A visual inspection of the casting indicated that there was no detectable increase in mold-metal reaction. This was confirmed by chemical analysis, which did not show any increase in oxygen or silicon over that encountered in lighter castings. It can be noted in figure 3 that the riser for the cube did not fill because of a short pour. The weight of the incomplete casting was 31 pounds.

The average R_A (Rockwell A scale) hardness for titanium cast in zircon molds was R_A 59 at the casting surface and R_A 40 on cut surfaces.

Chromite Rammed Molds

Sand tensile specimens made from Mouat chromite and cured at 480° F averaged 222 psi ultimate tensile strength. The sand mixing procedure was exactly the same as that used for rammed zircon sand molding.

Molds prepared from this mix and dried at 480° F were cast in the inductoslag and skull casting furnaces. Wedges, bells, and tees were successfully cast with and without zirconia mold washes. In all cases the castings,

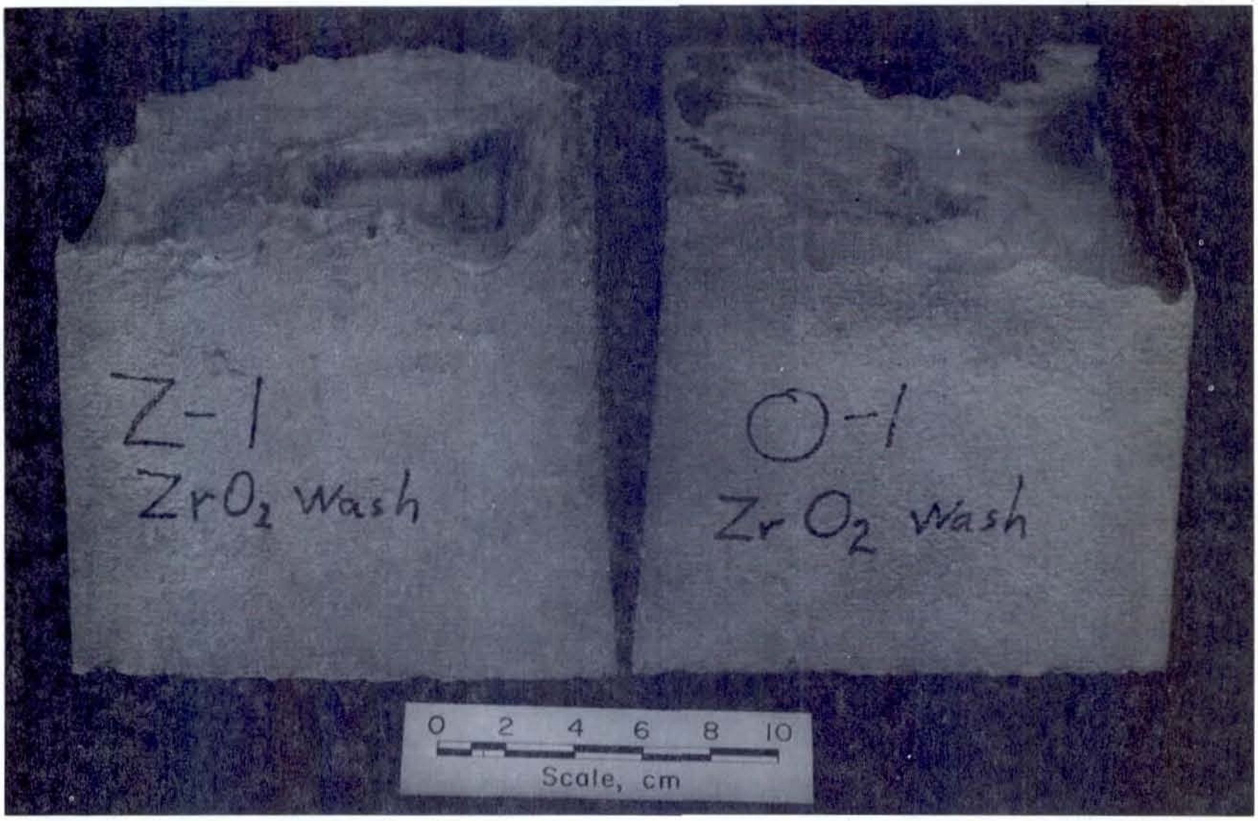

FIGURE 3. - Six-inch cubes cast in zircon (left) and olivine (right). The mold parting line was at the bottom of the cubes.

although free of internal porosity, had rougher surfaces than castings off the same patterns had when cast in zircon.

These castings were remade in purchased chromite sand with the same results. No significant difference could be detected between castings made in domestic submetallurgical-grade Mouat chromite and the purchased metallurgical grade chromite listed in tables 1 and 2.

The purchased chromite was used to make two test bar molds, which were dried at 480° F before pouring in the skull casting furnace. Both molds were poured in one heat, and six sound appearing bars were obtained. However, two bars from one casting were sawed off too short from the casting gate and could not be tested. One bar from the second mold was found to have a pinhole in the gage area during machining. Consequently, only three bars remained for mechanical testing. The mechanical results are shown in table 3, and the chemical content is given in table 4.

Molds of the 4-inch cube were made from Mouat and purchased chromite, washed with zirconia, dried at 480° F, and poured one at a time in the skull casting furnace. The results in all cases were unsatisfactory because of burn on due to excessive mold-metal reaction. A typical casting is shown in figure 4.

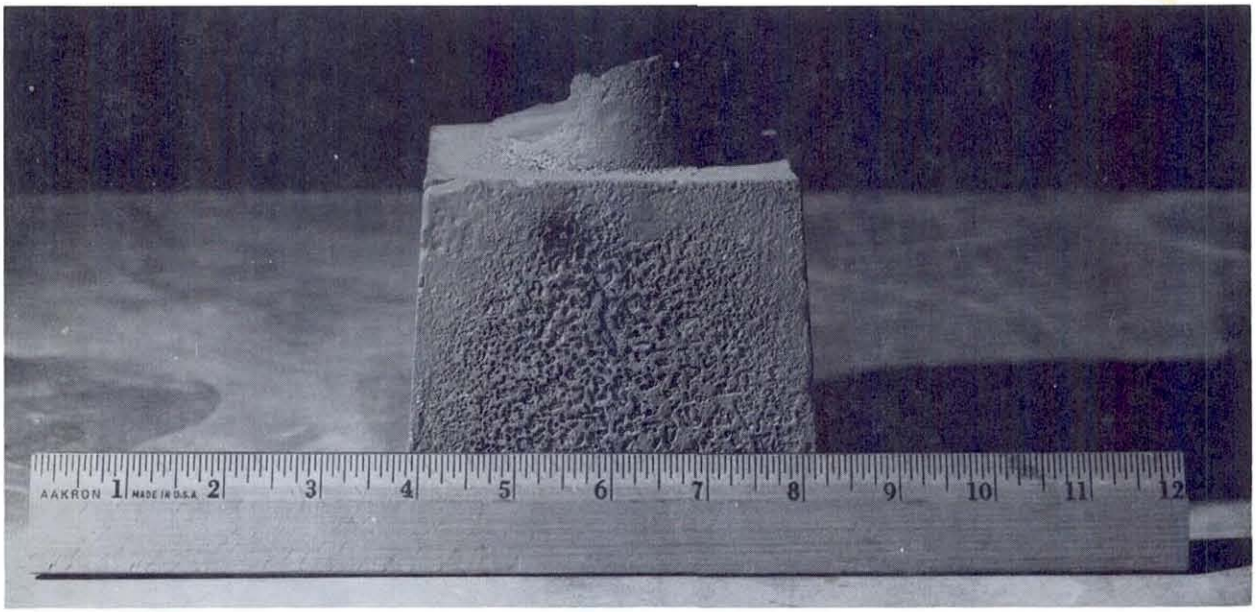

FIGURE 4. - Four-inch cube cast in zirconia-washed chromite mold showing that the metal has reacted with the mold material during casting.

Olivine Rammed Molds

Preliminary studies on the strength of waterglass-bonded olivine molding mixes indicated that a higher concentration of waterglass was needed for olivine than was required with zircon or chromite sands. The mix that was used for casting trials consisted of 5.4 pbw waterglass and 2.5 pbw water per 100 pbw of dry sand. Using this ratio, tensile specimens had an average strength of 135 psi when No. 90 olivine (tables 1 and 2) was used and 145 psi when No. 80 olivine was employed.

Bells, tees, wedges, and cubes were cast in molds prepared from No. 90 and No. 180 olivine mixes. It was found that commercial-grade castings could be produced in the inductoslag furnace off the bell, tee, and wedge patterns without using any mold wash. The 4- and 6-inch cubes were not inductoslag cast because the casting weights exceeded the melting capacity. The castings from olivine molds had smoother surfaces than castings poured in zircon molds. A typical casting is shown in figure 5. No. 90 olivine was the preferred molding sand because the casting surfaces were nearly as smooth as castings made in No. 180 molds, and the No. 180 molds tended to crack during the 480° F cure.

The only problem noted with castings poured in the inductoslag furnace was that about one of every three wedges cast had a hot-cornering defect in the drag at the juncture of gate and mold cavity. The defect, however, did not extend into the wedge body after the gate was cut off and the casting was ground. Figure 6 compares a typical olivine-cast wedge with one showing the defect.

FIGURE 5. - Typical inductoslag cast wedge poured in an unwashed olivine mold.

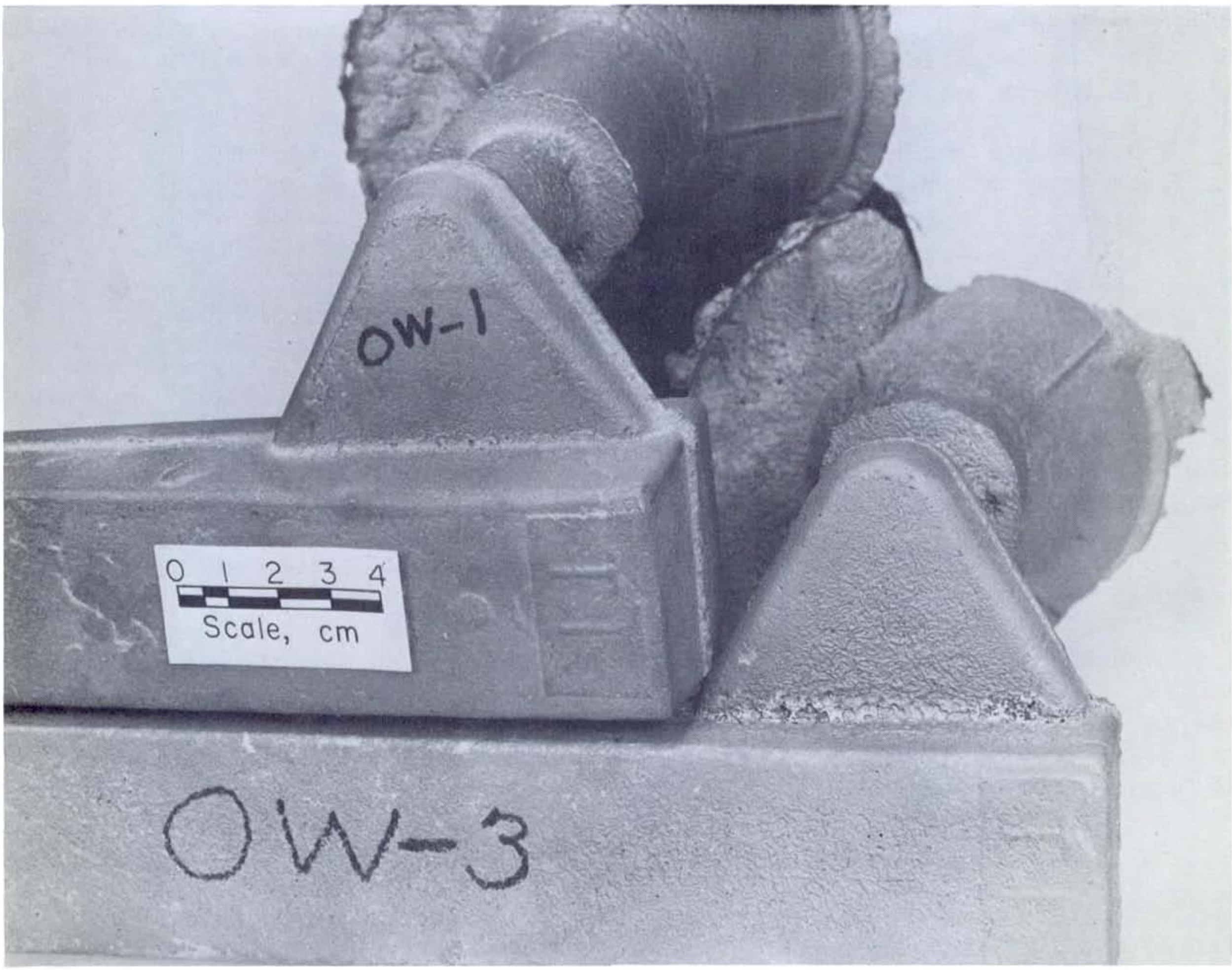

FIGURE 6. - Drag surfaces of two inductoslag cast wedges poured in unwashed olivine molds. Note hot-cornering defect on bottom casting at the juncture of gate and casting.

A surprise was encountered when attempts were made to produce wedge castings in the consumable-electrode arc-casting furnace. It was found that within a few seconds after the pour, molten metal blew back up the sprue, and hollow castings were obtained. Additional mold venting did not correct this problem. It was found, however, that a zirconia mold wash did solve the problem, and sound castings were made. The same results were obtained with bell, tee, and cube molds. That is, without the zirconia wash the castings were gas blown, but with a zirconia mold wash the castings were sound.

A 6-inch cube cast in No. 90 olivine is shown in figure 3. The mold was washed with 6 oz of zirconia slurried with 180 ml of 190-proof ethanol and 4 ml of 1:1 waterglass to water (2 ml of grade D waterglass dissolved in 2 ml of water). The washed mold was dried at 480° F. The weight of the short-poured casting was 31 lb.

Four test bar molds were made from No. 90 olivine and the usual amounts of water and waterglass. The unwashed molds were dried at 480° F and poured two at a time in the skull casting furnace. The first heat yielded only two sound test bars (both from the same mold). The other four bars had gas porosity. The next heat produced five sound bars and one with a gas-caused defect. The mechanical and chemical properties are given in tables 3 and 4, respectively.

The average hardness of the as-cast surface for castings made in olivine was R_A 65; hardness for cut surfaces averaged R_A 43.

Garnet Rammed Molds

Preliminary test on mold strength showed that garnet did not require more waterglass than zircon or chromite to produce strong molds that did not crack during drying at 480° F.

Sand tensile specimens prepared from M-80 garnet (tables 1 and 2) with 3.8 pbw waterglass and 2.5 pbw water per 100 pbw dry sand averaged 280 psi tensile strength.

Only the cored tee shape was cast in garnet molds because all of the castings had a very rough finish and gas voids in the cope metal.

Rough surfaces and gassy copes persisted even when mold washes and increased venting were used. A finer garnet sand, M-100, which had an American Foundryman's Society grain fineness No. (AFSgfn) of 100 was tried alone and in blends with M-80 (AFSgfn 50), but no improvement was noted in the quality of castings produced.

SUMMARY OF RAMMED MOLDS

Of the four sands studied (zircon, chromite, olivine, and garnet) only zircon and olivine were capable of making industrially acceptable castings at the 6 x 6-inch cross-sectional size. Chromite was acceptable at the

1-3/4 x 1-3/4-inch cross-sectional level, but it suffered from extensive burn on at the 4- x 4-inch cross-sectional size. Garnet was unacceptable at all casting thicknesses because of gassy cope metal and very rough casting surfaces.

Samples for electron microprobe analyses were prepared from the 1-3/4 x 1-3/4-inch ends of titanium wedges inductoslag cast in unwashed zircon, olivine, and Mouat chromite molds in an effort to obtain some insight on the mold-metal reactions taking place with each sand.

The scanning electron micrographs for the zircon-titanium interface showed zircon grains at all locations. The larger zircon ($ZrSiO_4$) grains were surrounded by zirconium oxide (ZrO_2) with a high silicon (Si) low zirconium (Zr) layer of titanium about 5 micrometers (0.0002 inch) thick between the ZrO_2 and the titanium (Ti). Semiquantitative analysis of this layer suggests a suboxide of Ti and Si because the combined Ti and Si content is too low for a silicide and too high for the silicate. Silicon contamination was detected to a depth of about 0.020 to 0.050 inch. These results point strongly toward the thermal dissociation of some of the $ZrSiO_4$ into ZrO_2 and SiO_2 with the silica being reduced by Ti to SiO or Si accompanied by the formation of one or two molecules of TiO, with subsequent dissolution of the products in molten titanium. However, when the casting forms a frozen shell the reaction ceases, with the result that contamination is slight and relatively shallow in depth.

The electron microprobe scan of the chromite-titanium interface showed reaction to a depth of about 0.10 inch. A curious development is that the outermost metallic layer is relatively pure titanium for a depth of about 0.002 inch before reaching a two-phase region consisting of Ti and a solid solution of iron (Fe) and chromium (Cr) and Ti. The solid solution phase contains roughly 5 pct Fe and 7 pct Cr. The Cr:Fe ratio of the solid solution is very close to the Cr:Fe ratio of Mouat chromite, which indicates that the oxides of both Cr and Fe are reduced to about the same extent by molten titanium. The nearly pure Ti at the surface and the absence of magnesium and aluminum there and in the interior indicate that the viscosity of the solid solution phase (Fe and Cr in Ti) is greater than the viscosity of the Ti phase, and that the outermost Ti layer froze against a surface that was predominately magnesium and aluminum oxides, with minor quantities of iron and chromium oxides.

The electron microprobe scan of CP titanium (commercially pure Ti) inductoslag cast in unwashed olivine showed that contamination of the sample was very slight. A different microstructure was noted near the surface, but it did not appear to be contamination from mold-metal reaction. However, when Ti-6Al-4V alloy was inductoslag cast in an unwashed olivine mold, it was found that Si had diffused into the matrix Ti for a distance of 0.004 inch and Fe was detected in a grain boundary phase to a depth of 0.010 inch. Thus, it appears that mold-metal reaction in olivine molds consists primarily of reduction of iron and silicon oxides by titanium. However, the extent of reaction is slight when the molds are poured in the inductoslag furnace.

Waterglass-bonded olivine molds that were poured in the skull casting furnace did not produce good castings unless a zirconia mold wash was used. Without a protective wash the castings were subject to gas voids. Physical and chemical analyses of gas blown castings failed to reveal the mechanism of the mold-metal reaction that caused this problem. However, the problem can be solved by lightly spray coating the molds with water or ethyl alcohol slurries of 80 pct minus 325-mesh monoclinic zirconia and drying the molds at 480° F prior to pouring.

SHELL MOLDS

Shell Patterns

Three patterns were available for shell molding. The patterns made of aluminum and brass, are illustrated in figures 7-9. The largest of these patterns was the cored cylinder, shown in figure 7, which had an outside diameter of 3-1/2 inches, an inside diameter of 2-3/8 inches, and a length of 5 inches. The weight of the raw casting was 8 lb, and the finished casting weight was 4 lb.

Zircon Shell Molds

The zircon shell molding tests were divided into two basic groups. The first group of shells was made from novalak-coated zircon, and the second group made from waterglass-coated zircon.

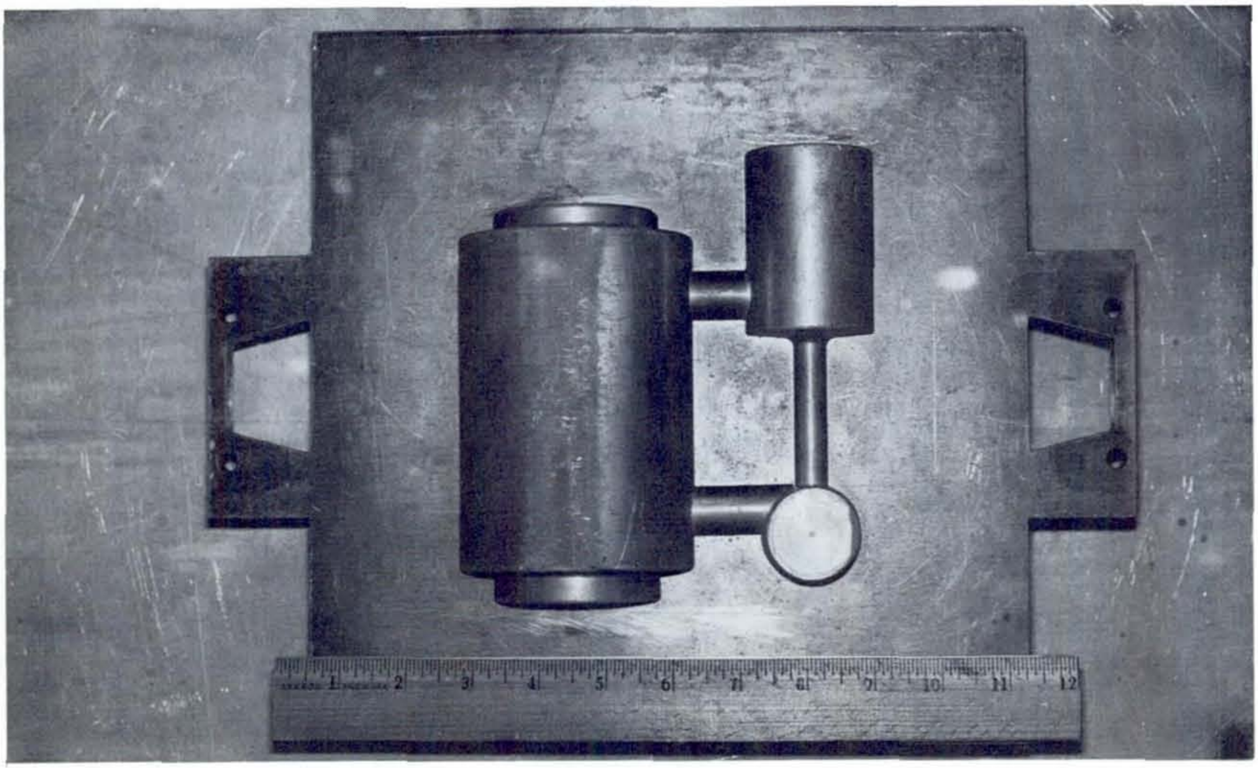

FIGURE 7. - Shell molding pattern for cored cylinder.

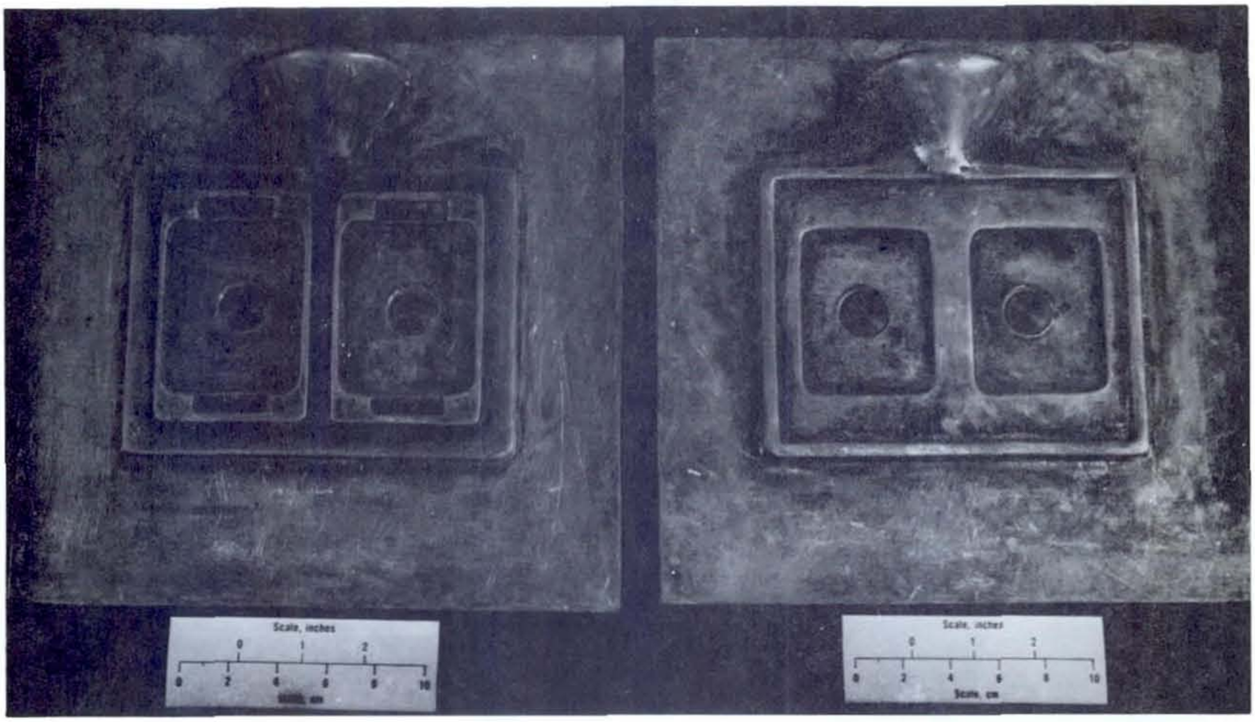

FIGURE 8. - Shell molding pattern for wall plate. Front and back views.

The novalak coating was applied to the zircon by dry mixing the sand with powdered phenol-formaldehyde resin and powdered hexamethylenetetramine (hexa) in a rotary cement mixer for 1 minute before slowly adding a measured amount of 190-proof ethyl alcohol. The mixing was continued until the sand blend first became plastic (about 1 minute) and then broke down to minus 20-mesh particles (about 12 minutes). The coated sand was then discharged through a 20-mesh screen and air dried overnight before using.

A range of resin:hexa:zircon ratios was investigated by preparing molds off the wall plate pattern shown in figure 8. But it soon became apparent that the lowest concentration of resin that would still produce shells with the requisite strength for handling and pouring produced the best castings.

This mix had the following composition: 100 pbw zircon, 3 pbw powdered phenol-formaldehyde resin, 0.75 pbw powdered hexa. The mix was coated by slowly adding 0.60 pbw of 190-proof ethyl alcohol to the rotary cement mixer and proceeding as given previously.

Molds were prepared from this mix off the wall plate and the cylinder pattern. The patterns were heated to 540° to 570° F and inverted on the dump box for 4 minutes. The shells were then oven cured on the patterns for 4 minutes at 600° to 640° F before stripping. After the mold halves were cemented together, the molds were cured at 500° F for 30 minutes before pouring with titanium.

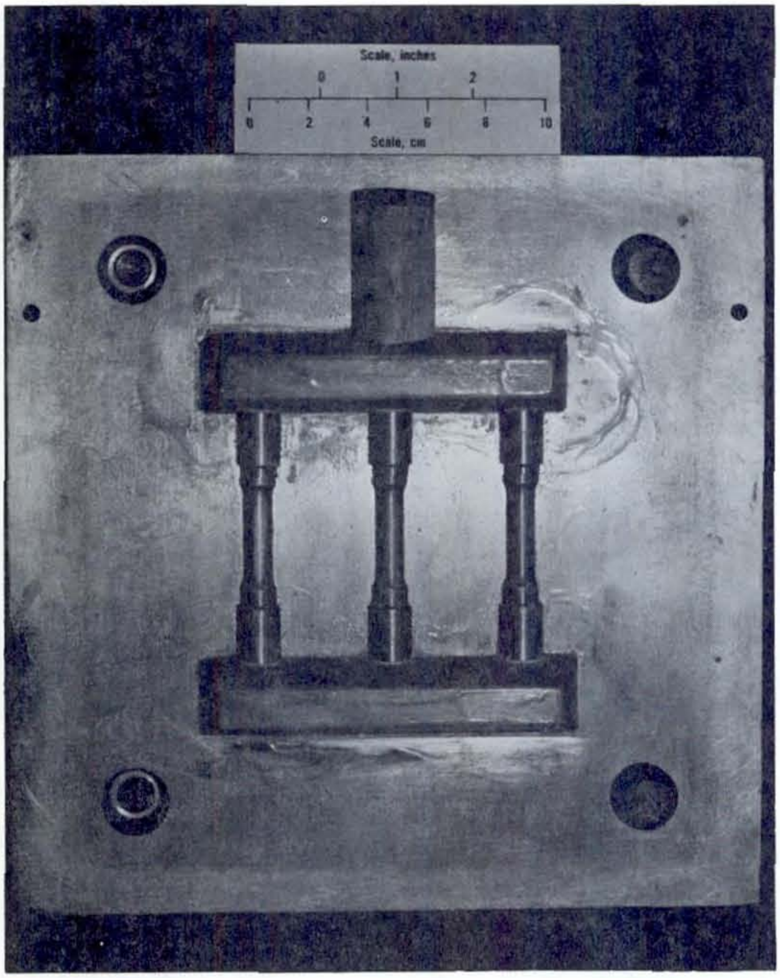

FIGURE 9. - Shell molding pattern for 1/4-inch test bars.

A further complication arose after pouring from residual organic volatiles remaining in the inductoslag furnace. Several backfills and pumpdowns were necessary to remove noxious volatiles from the system after each pour. The best casting produced in the first series is shown in figure 10.

The second series, waterglass-coated zircon, was more successful than the first. A preliminary series of sand mixing tests led to the following procedure for forming shells: Zircon sand (AFSgfn 94) was mixed in the rotary mixer with 3 wt-pct zircon flour (60 pct minus 325 mesh) for 2 minutes dry, then 3.8 pbw of grade D waterglass was added and mixing was continued for 8 minutes longer before discharging. The discharged mix was put in the dump box immediately.

The molding procedure consisted of the following steps:

1. The pattern was heated to surface temperature of 465° F in an electric oven.

2. The pattern was removed from the oven and clamped on the dump box.

3. An audible frequency air vibrator attached to the bottom of the dump box was activated, and the dump box was inverted.

4. Air vibration was discontinued after 2 seconds of inversion.

5. The dump box was returned to the upright position after 4 minutes of inversion.

6. The pattern was removed from the dump box and placed in an electric oven set at 480° F for 2 minutes.

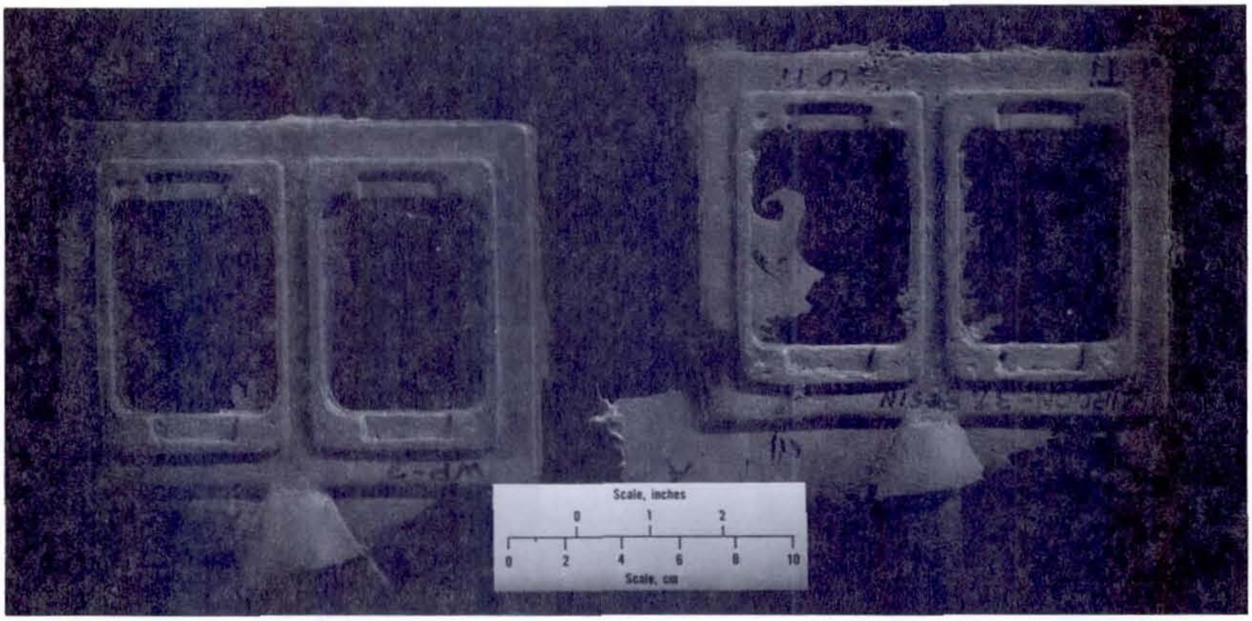

FIGURE 10. - Shell cast wall plates. The left casting was poured in waterglass-bonded zircon and the right casting in novalak-bonded zircon.

7. The shell was removed from the pattern.

8. Mold halves were pasted together with zircon cement.

9. Molds were cured at 480° F for an hour.

It was found that slightly superior mold detail could be obtained with zircon-waterglass shell sands (compared to novalak-zircon shells) off the wall plate pattern when the air vibrator was used, but without the air vibrator novalak-coated zircon produced better pattern detail. It was also found that zircon-waterglass molds were easier to strip from the pattern. Most importantly, it was found that the zircon-waterglass shells produced acceptable castings. A typical waterglass-coated zircon casting is compared in figure 10 with the best novalak-coated zircon casting produced in this research.

The mechanical data for shell cast test bars and the chemical analyses are given in tables 5 and 6, respectively.

TABLE 5. - Mechanical data of titanium Inductoslag castings in zircon, chromite, and olivine shell molds

Heat No.	Test bar No.	Ultimate tensile strength, ksi	Yield strength (0.2 pct offset), ksi	Elongation (in 1 inch), pct	Reduction in area, pct
			ZIRCON		
1	1	62.1	52.2	32	58
1	2	62.0	50.9	37	61
1	3	63.0	50.9	34	59
			MOUAT CHROMITE		
2	1	[1]106.3	[1]90.3	21	28
2	2	107.0	91.7	22	32
2	3	([2])	([2])	([2])	([2])
			OLIVINE		
3	1	64.1	51.3	31	54
3	2	64.5	51.5	29	53
3	3	65.1	52.0	31	46

[1] The unusually high strength is the result of high oxygen content in the melt stock.

[2] Specimen failed post-test examination.

TABLE 6. - Chemical analyses of test bars listed in table 5

Sand	Heat[1] No.	Concentration ppm				
		O	H	N	Cu	C
Zircon..................	1	1,350	40	54	100	500
Mouat chromite...........	2	[2]3,320	26	172	200	490
Olivine..................	3	1,060	140	150	500	130

[1] Heat No. refers to table 5.

[2] The unusually high oxygen content was due to using oxyacetylene torch cut revert for melt stock.

Chromite Shell Molds

Because of the lack of success with resin-bonded zircon shell molds for casting titanium, only waterglass-bonded chromite shells were investigated.

Preliminary tests indicated that 3.8 pbw waterglass was the lowest concentration of waterglass that could be used per 100 pbw dry sand to make molds of the required strength for pouring. However, to obtain this strength 0.5 pbw of water per 100 pbw sand had to be added to the mix. The water addition made the 3.8 pbw waterglass shells equivalent in strength to shells made from 5.4 pbw waterglass and no additional water.

The molding mix was prepared in 45-lb batches in the rotary mixer from 100 pbw Mouat chromite and the addition of 0.5 pbw water dissolved in 3.8 pbw waterglass. After the liquid addition the mix was rotated for 10 minutes. The discharge was loaded into the dump box immediately.

Shells were made off the patterns shown in figures 7, 8, and 9. The molding sequence was nearly the same as that used for zircon-waterglass mixes; the difference was that 4 seconds of vibration after inverting the dump box (instead of 2 seconds) produced the best shells with regards to mold detail and strength.

The castings produced in chromite molds were not as good as those produced in zircon shells. There was a tendency toward surface defects such as pinholes and surface roughness from minor burn on (mold-metal reaction). Burn on is shown in figure 11, which is a closeup photo of a test-bar casting.

The mechanical properties of cast test bars are given in table 5 and the chemical content in table 6.

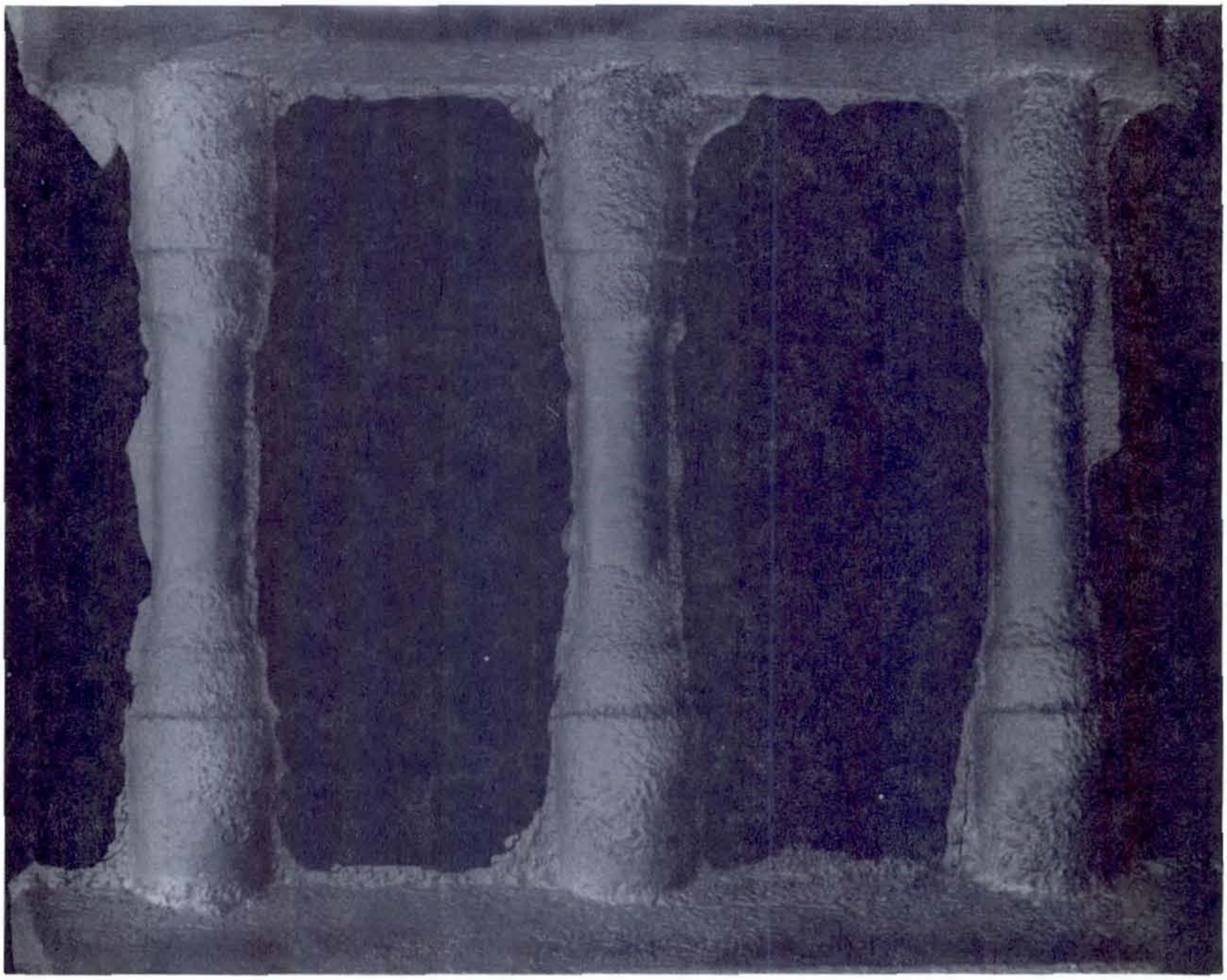

FIGURE 11. - Shell cast test bars from a waterglass-bonded chromite mold showing that the metal has reacted with the mold material during casting.

Olivine Shell Molds

Preliminary tests with olivine shells bonded by waterglass also showed that some water was needed in the basic mix in order to form strong shells. The mixing and molding procedure was the same as for chromite except that the molds required more waterglass and water to form shells of comparable strength. The mix used to make the best molds and castings was composed of 65 pbw No. 90 olivine, 35 pbw No. 180 olivine, and 5.4 pbw waterglass dissolved in 1.0 pbw water.

It was found that a zirconia mold wash improved the casting finish, although acceptable castings off the wall plate and test bar patterns were made without washes. However, acceptable castings could not be made off the cylinder pattern without a zirconia wash. Typical olivine shell castings are shown in figures 12, 13, and 14. A radiograph of an olivine shell-cast cylinder is shown in figure 15.

Olivine test bars were cast in shell molds without washes in the inductoslag furnace. The mechanical and chemical evaluations are given in tables 5 and 6, respectively.

Because the olivine tests appeared to be quite promising, two series of sand tests were run to study the effects of different mesh sizes and varying quantities of waterglass on the tensile strength of the shells produced. The procedure for making shell sand tensile specimens and the method for determining tensile strength is given in appendix A.

The first series studied blends of No. 90 and No. 180 olivine sands with 5.4 pbw waterglass and 1.0 pbw water per 100 pbw of blended sands.

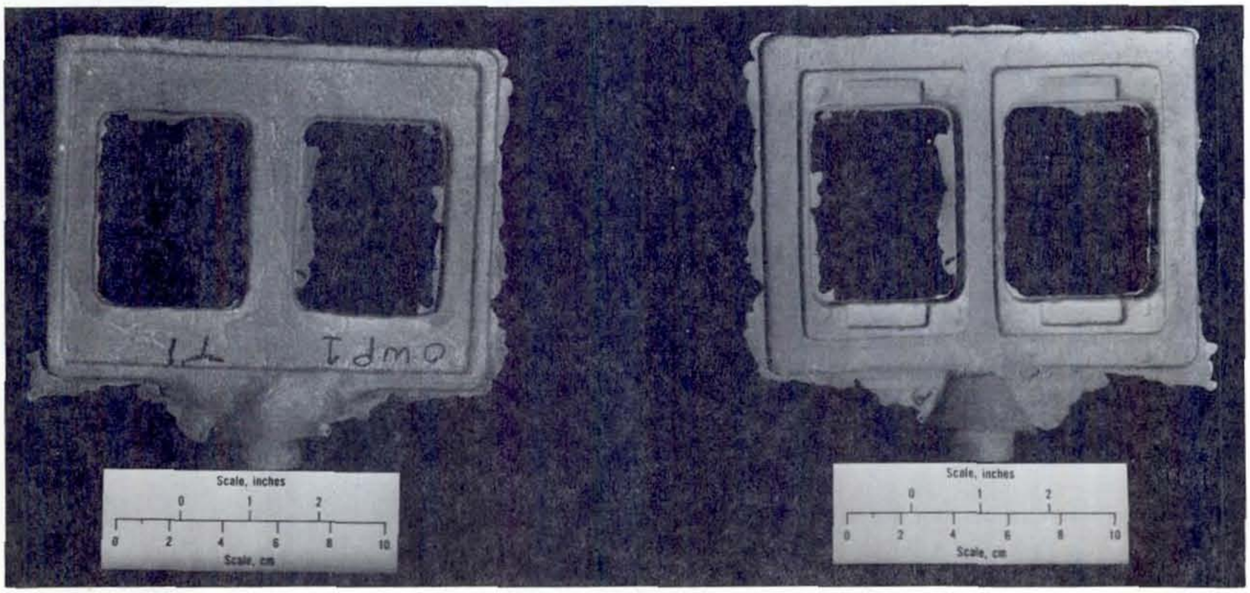

FIGURE 12. - Typical wall plate shell casting from an unwashed waterglass-bonded olivine mold (front and back views).

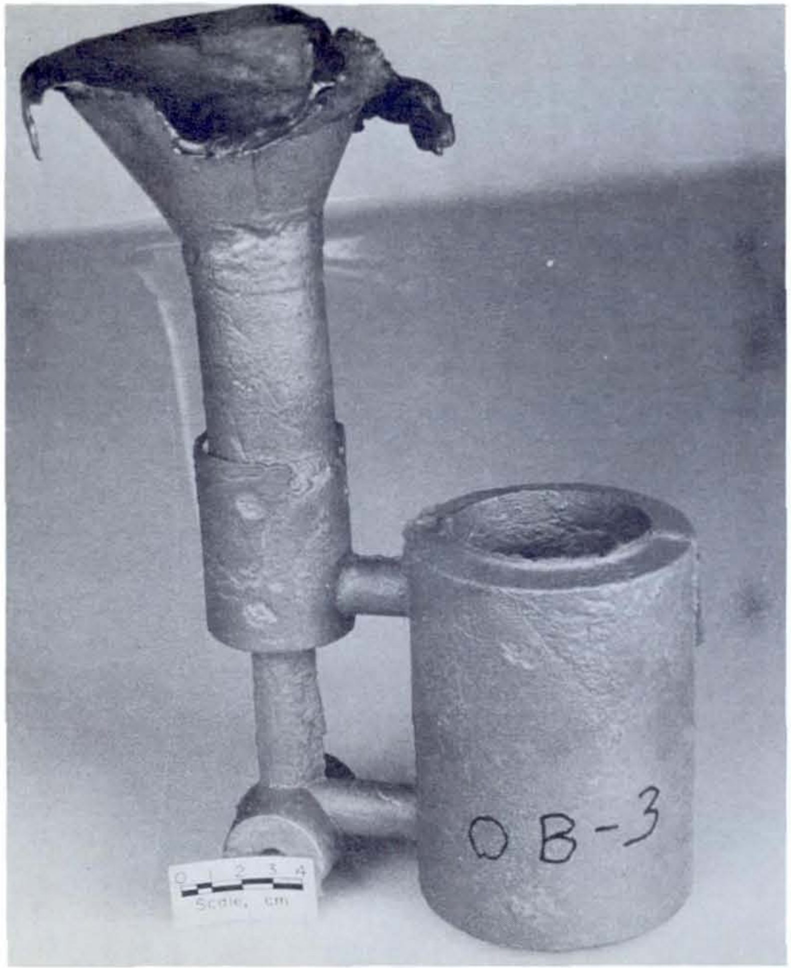

FIGURE 13. - Side view of a shell cast cored cylinder from a waterglass-bonded olivine mold.

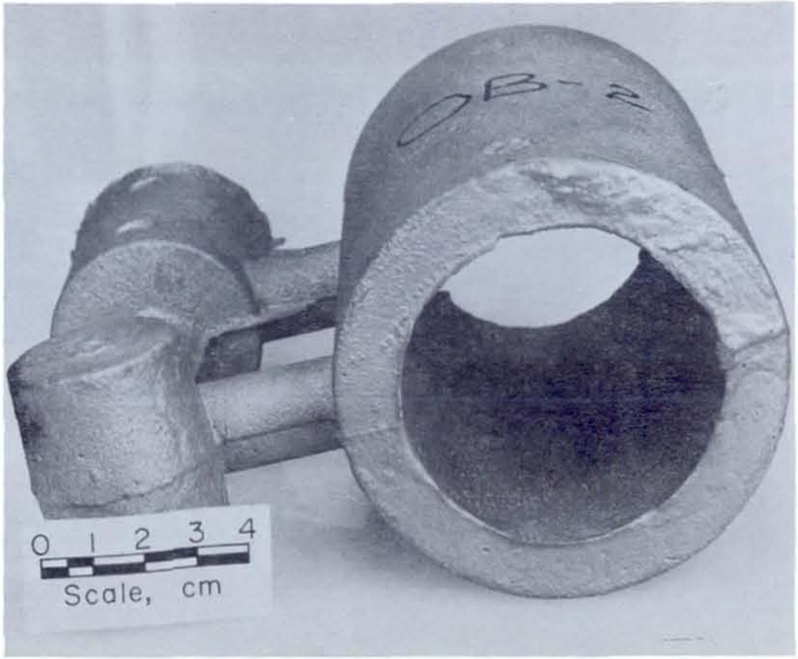

FIGURE 14. - Bottom view of a shell cast cored cylinder from a waterglass-bonded olivine mold.

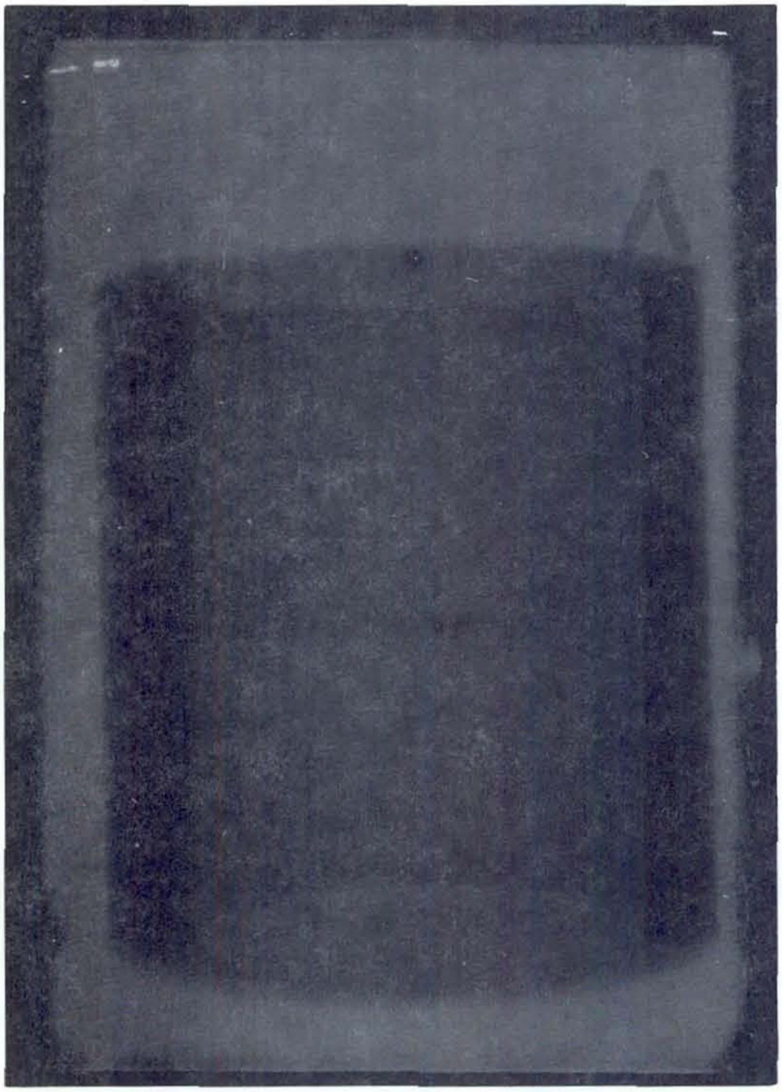

FIGURE 15. - Radiograph of a shell cast cored cylinder from a zirconia-washed waterglass-bonded olivine mold.

The results, shown in table 7, indicate that sand particle size has little effect on the strength of the molds under test conditions. Therefore, No. 180 olivine sand would generally be used because it will give a smoother finish to a casting. However, if greater permeability of the mold is desired, rather than surface smoothness, No. 90 olivine can be used.

The second series of tests was designed to study the effect of waterglass concentration on mold strength with a constant concentration of water (1.0 pbw water per 100 parts of No. 180 olivine). The results, listed in table 8, indicate that mold strength tends to increase with increasing waterglass concentration in the range studied. However, because the fusion point of the sand mix decreases with increasing waterglass concentration, it would be prudent to use the lowest concentration of waterglass that will give enough strength for mold handling and pouring.

Summary of Shell Molding

Of the three sands studied, there is little doubt, on the bases of visual, radiographic, and chemical examinations, that waterglass-bonded zircon provides the most refractory shell molds for casting titanium. Olivine is the second best sand with regard to resisting attack by molten titanium, but on castings of larger cross section it may prove difficult to hold dimensions without backing the shells because the shells tend to become plastic when overheated. Chromite, on the other hand, does not become plastic at pour temperatures, but it is subject to mold-metal reaction even with small cross-section castings.

TABLE 7. - Tensile strength of blended olivine shell[1]

Composition		Tensile strength		
No. 90 olivine, pbw	No. 180 olivine, pbw	Average,[2] psi	Maximum psi	Minimum, psi
100	0	137	173	118
75	25	127	191	82
50	50	104	138	87
25	75	132	146	124
0	100	134	156	98

[1] All mixes contained 5.4 pbw grade D waterglass and 1.0 pbw water per 100 pbw of dry sand.

[2] Average of 6 tests.

TABLE 8. - Tensile strength of No. 180 olivine specimens with various concentrations of waterglass[1]

Waterglass, pbw/100 parts olivine	Tensile strength, psi		
	Average[2]	Maximum	Minimum
3.1	88	101	70
3.8	76	84	66
4.6	125	162	90
5.4	134	156	98
6.1	208	271	147

[1] All mixes contained 1.0 pbw water per 100 pbw dry olivine.

[2] Average of 6 tests.

No problems were encountered in forming excellent shells on shallow draw patterns with any of the sands, but a shadow (uneven packing of sand) generally developed on deep draw shells with all three sands. It is believed that this fault could be corrected if the sand was air blown onto the pattern instead of dumped. Many modern commercial shell molding machines, and most shell core molding machines, air blow the molding mix onto the pattern in order to achieve even packing (to avoid shadows).

CONCLUSIONS

Waterglass-bonded zircon rammed or shell molds have potential for commercially casting titanium. However, more research is needed on shell molding. It was demonstrated that commercial-grade castings up to 8 lb can be made in shell molds, and castings up to 31 lb can be poured in rammed molds. A zirconia mold wash is beneficial for castings of 4-inch square, or larger, cross section.

The statement made for zircon sand also applies to olivine rammed and shell molds, but a zirconia wash is mandatory for olivine molds of all sizes when the molds are poured in the skull casting furnace.

The behavior of rammed olivine molds in the inductoslag furnace differs considerably from their behavior in the skull casting furnace.

For inductoslag melting, it was found that casting weights up to the crucible capability of 8 lb of titanium could be cast in unwashed molds without getting noticeable mold-metal reaction. In the skull casting furnace, however, it was found that no sound castings, regardless of size, could be produced without a zirconia mold wash. The reason for this marked difference in behavior has not been ascertained.

Chromite and garnet sands are subject to extensive mold-metal reaction with molten titanium, and they do not appear to be worthy of further study.

The most serious problem of waterglass-bonded rammed olivine or zircon molds is the lack of green strength. The green strength of molds and cores is so low that it would be difficult to get large molds or cores into a drying oven without cracking or distorting them during handling. Research should be done on developing green strength in the molding mixes without detracting from the quality of titanium castings produced.

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APPENDIX A.--SAND TESTING PROCEDURES

Shell Tensile Specimen Preparation

There is no accepted specification for preparation of thermal-set, waterglass-bonded shell tensile specimens. Therefore, a procedure was devised so that data could be obtained for internal comparative purposes. The procedure was based on the tentative standard for hot shell tensile strength given by the AFS (American Foundryman's Society).[1] The AFS procedure was designed for organic coated sands. Consequently, some modifications were required to suit the characteristics of waterglass-bonded sands. The detailed procedure was as follows: Spray the three 1/4-inch-thick specimens gang mold, shown in figure A-1, with silicone mold release. Heat the gang mold to at least 570° F surface temperature in an electric oven set at 660° F. Remove the gang mold from the oven and mount it on the steel plate with an attached audible frequency air vibrator. Let the gang pattern cool to a surface temperature of 525° F. Then, with the air vibrator on, dump the hopper strike-off assembly (shown in fig. A-1). Keep the air vibrator on for 2 seconds after the dump. Then reverse the hopper strike-off assembly and flatten the heaped sand in the gang mold. Strike the molds level and put the gang pattern in a 660° F oven. After 5 minutes remove gang pattern and note surface temperature; it should be 525±20° F. Remove specimens from the pattern and, after cooling, seal in plastic bags for storage until tensile testing.

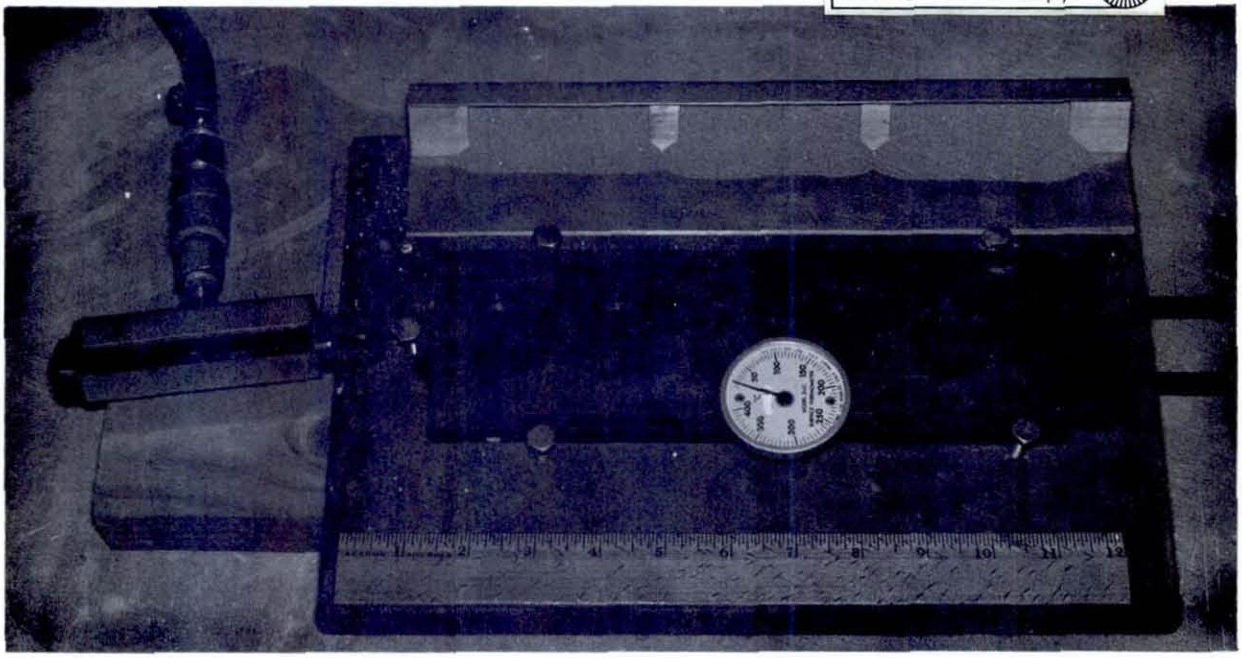

FIGURE A-1. - Shell tensile specimen gang pattern assembly with hopper strike-off assembly (top).

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[1] American Foundrymen's Society. Molding and Core Test Handbook. Des Plaines, Ill., 7th ed., 1963, pp. 15-1 to 15-4.

The tensile test results for a given sand were reproducible within ±25 pct, if the specimens were prepared by the same operator. Thus, the results were useful for comparative purposes. However, the reproducibility of results was greatly dependent on the operator. For instance, specimens from replicate mixes were prepared by three different operators, and two of them made specimens with a spread of less than ±25 pct of the average tensile strength for either operator, but the third operator's specimens were 10 to 57 pct above the average tensile strength obtained by the first two operators. The reason for the large deviation was that the third operator used greater pressure on the slightly heaped sand when tamping the gang mold before strike off. Therefore, for comparative purposes of various mixes, the specimens should be prepared by the same operator.

Rammed Sand Tensile Specimen Preparation

The preparation of rammed sand tensile specimens followed exactly the procedure given by the AFS for determination of baked tensile strength of cores (briquet method).[2] The specimens were rammed in a two-part aluminum core box with a loading hopper by three blows of a standard 14-lb rammer. The equipment is shown in figure A-2. The green specimens were cured for 1 hour at 480° F, cooled to room temperature, and sealed in plastic bags to await tensile testing.

Both the 1-inch-thick rammed sand and the 1/4-inch-thick shell specimens were pulled on a universal testing maching at a loading rate of 600 lb-per-min at room temperature. The specimens were gripped with the briquet clips specified in ASTM specification C 190-72.

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[2] American Foundrymen's Society. Molding and Core Test Handbook. Des Plaines, Ill., 7th ed., 1963, pp. 13-1 to 13-5.

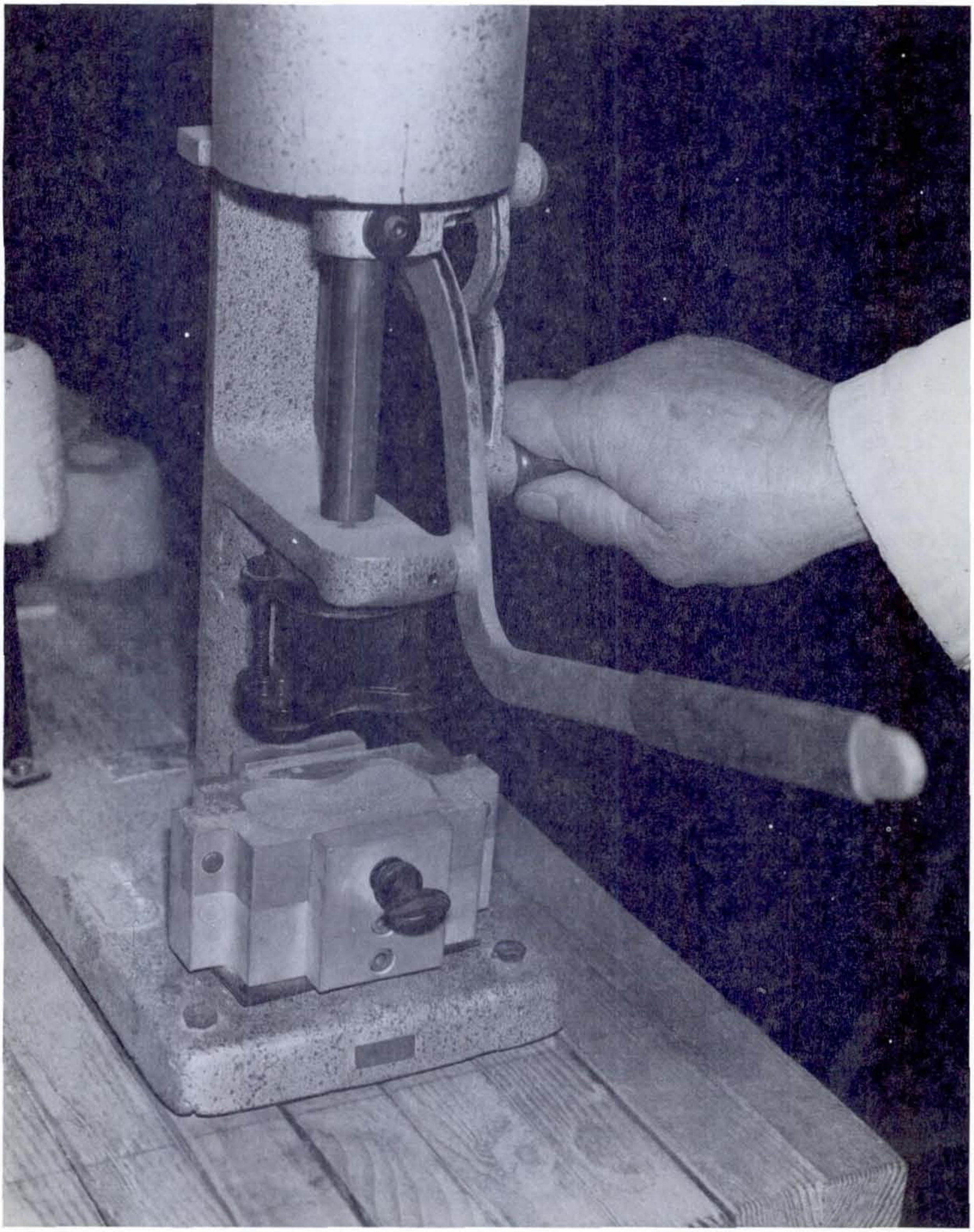

FIGURE A-2. - Briquet mold assembly and 14-lb rammer for preparation of rammed sand tensile specimens.

APPENDIX B.--MECHANICAL TEST PROCEDURE

To demonstrate that the molding and casting processes examined in this study did not adversely affect the mechanical properties of cast titanium, the specimens were prepared and tested as nearly as possible in accordance with the American Society for Testing and Materials standard ASTM B 367. ASTM B 367 sets up certain standards for the testing of the cast material covered by it. According to this specification the following must be adhered to:

> "8.2 tension test specimens shall be machined and tested in accordance with ASTM Method E8, Tension Testing of Metallic Materials. Tensile properties shall be determined using a strain rate of 0.003 to 0.007 in/in/min through the specified yield."

ASTM Method E8 states that after the material under test passes through yield, the strain rate may be increased to a rate of up to 0.05 in/in/min, but this specification does not require such action.

Because of equipment limitations, we were not able to reach the 0.003 to 0.007 in/in/min strain rate requirement set forth in ASTM B 367; the closest we were able to come to this requirement was a strain rate of 0.01 in/in/min. Since the changing of strain rate indicators on the test machine in use on this project requires interrupting the test, and since the requirements of ASTM B 367 only suggest a change in strain rate, the single strain rate used throughout each test is 0.01 in/in/min.

The specimen configuration chosen for this program is a standard 0.250-inch-diameter threaded tensile specimen. The tensile specimen, shown in figure B-1, conforms to the applicable ASTM recommendations.

Testing was carried out on a 60,000 lbF (27,200 kgF) Baldwin Universal Testing Machine equipped with an MA-1 Stress-Strain Recorder.[1]

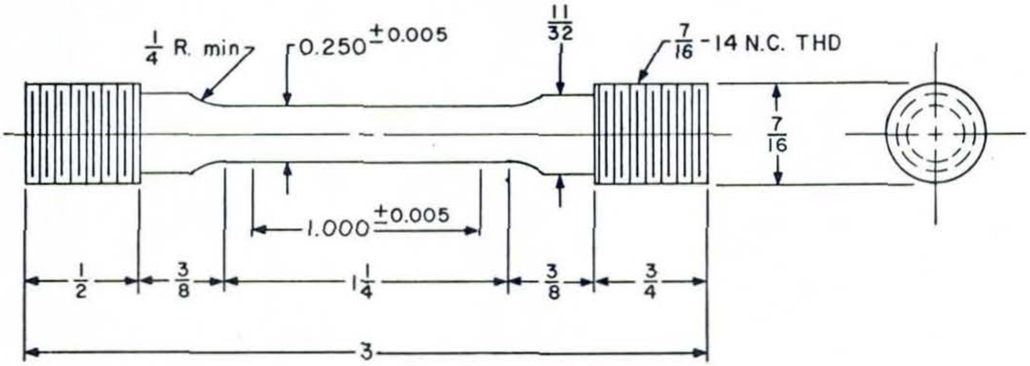

FIGURE B-1. - Standard configuration of cast titanium tensile specimens.

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[1] Reference to specific equipment does not imply endorsement by the Bureau of Mines.

PROCEDURE

Each tensile specimen or lot of tensile specimens was cast to shape using the appropriate casting method and mold. After casting the tensile specimens were individually finish machined to conform with the configuration shown in figure B-1. After finish machining, each specimen was marked with an identifying test number and dye-penetrant inspected for defects. Careful notes were kept on any discontinuities found during the inspection, and any results from the flawed specimens were discarded.

After the inspection of the specimens, they were then gage marked and prepared for tensile testing. The gage-marked tensile specimens were placed in the grips of the testing machine, and the extensometer was attached to the specimen and the stress-strain recorder. The tensile test was then performed in the manner mentioned previously, and a post-test examination of the specimens was made. Any results from specimens failing either the dye-penetrant examination before testing or the post-test examination were discarded.

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Shape-Casting Titanium in Olivine, Garnet, Chromite, and Zircon Rammed and Shell Molds

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16. Abstract (Limit: 200 words)

In seeking substitutes for such critical metals as chromium, cobalt, and nickel, the Bureau of Mines investigated techniques for shape-casting titanium in rammed sand molds. Castings were made in olivine, garnet, chromite, and, for comparison, zircon. It was found that commercial-grade castings up to 31 lb (maximum capacity of furnace) could be made in zircon or olivine molds if a zirconia mold wash was used. Castings up to 4 lb could be made in chromite molds, but heavier castings suffered from some mold-metal reaction. Garnet molds were found to be unsatisfactory for castings of all sizes because of gas blows and rough surfaces. In other tests shell castings of acceptable quality were produced in water-glass-bonded zircon and olivine molds up to weights of 8 lb, but chromite molds were unsatisfactory because rough casting surfaces were caused by mold-metal reaction. Unlike the currently used commercial processes, neither the rammed-sand nor the shell-molding processes developed by the Bureau of Mines generate noxious fumes at any step.

17. Document Analysis a. Descriptors

Titanium castings
Shell molding
Molding techniques
Molding materials
Foundry sands
Binders

b. Identifiers/Open-Ended Terms

Titanium
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Olivine
Garnet
Zircon
Chromite

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