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PRELIMINARY STUDY INTO SHELL MOLD CASTING OF NOMINAL 60-NITINOL ALLOY

William J. Buehler, et al

Naval Ordnance Laboratory
White Oak, Maryland

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PRELIMINARY STUDY INTO SHELL MOLD CASTING OF NOMINAL 60-NITINOL ALLOY

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NAVAL ORDNANCE LABORATORY
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PRELIMINARY STUDY INTO SHELL MOLD CASTING OF NOMINAL 60-NITINOL ALLOY

For many years there has been a continuing program at NOL to improve the quality of EOD tools. This present study was initiated to determine the feasibility of shell mold casting nominal 60-Nitinol into suitable EOD tools. This investigation has been performed as a part of task number ORD 35C-502/081-1/UF 34-373-502.

ROBERT WILLIAMSON II
Captain, USN
Commander

W. Scanlon

W. W. SCANLON
By direction

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INTRODUCTION

1. 60-Nitinol is a generic name for an alloy containing 58 to 62 weight percent nickel, the remainder of the alloy being titanium. These alloys, in spite of their high nickel content, are stably nonmagnetic. Further, because of a retrograde solid solubility between the TiNi and $TiNi_3$ phase fields they are capable of being precipitation hardened to very high hardness levels.

2. The combination of high hardness coupled with the nonmagnetic feature made these materials ideally suited to EOD application. As a result some effort has been expended in determining the most effective way in which to reduce the nominal 60-Nitinol into suitable tool shapes.

3. Initial efforts were concentrated on closed-die forging, the method generally employed in making most steel tools. Forging nominal 60-Nitinol into tool shapes was found to be both difficult and costly. While forged 60-Nitinol tools were possible, their potential high cost dictated the use of alternate fabrication methods.

4. A comparison of tensile and hardness properties for both wrought[1] and arc-cast[2] 60-Nitinol are given in Table I. These data, while liberally averaged, tend to indicate only minor property degradation associated with the cast structure. The most noteworthy exception lies in the tensile elongation of the "furnace cooled" hot wrought material. Some concern might be raised over the relatively low tensile elongations (strain-to-failure) in both cast and wrought materials. However, the property of "toughness" is probably a more important criterion than tensile elongation for most tools. In spite of the relatively low strain-to-failure for 60-Nitinol, the toughness (unnotched) value of 38 ft-lbs[1] for wrought material in the quench-hardened ($\sim 60\ R_C$) condition is of suitable magnitude. Based upon these and other property data, efforts were redirected into the possibility of "casting" tool shapes.

5. Knowing that prealloyed TiNi-base alloys were not reactive with graphite,[3] up to about 200°C over the melting temperature ($\sim$1500°C), provided a basis for a Nitinol tool casting program. Initial efforts consisted of vacuum induction melting a 58-Nitinol alloy in an ATJ graphite crucible and pouring the molten alloy into a graphite mold. The results of efforts to cast a 60-Nitinol hammer head were reported previously.[4] Based upon this study it was concluded that casting nominal 60-Nitinol tools in a graphite or graphite-faced mold was feasible. However, molding design and associated costs would be prime factors in the suitability of this technique.

6. With the cost factor in mind, moldings of silica with a thin graphite layer or barrier were considered. Green sand molding was eliminated for obvious reasons, uppermost being the dimensional control of the cast part. Both precision casting ("lost wax") and shell molding methods were appraised. The former was downgraded in priority because of its complexity and cost. By elimination shell molding was chosen. It was less complex, provided adequate dimensional accuracy and produced a suitable cast surface finish. The major question surrounding shell molding was its compatibility with the nominal 60-Nitinol material. The present report summarizes the initial efforts in shell mold casting of 60-Nitinol. A more complete description of the shell molding process is given in Appendix A.[5]

EXPERIMENTAL PROCEDURE

7. In order to conduct a meaningful experiment it is necessary to both develop required information and anticipate potential problem areas. Devising an experiment around the shell mold casting of nominal 60-Nitinol was no exception. Certain key results had to be favorable in order for shell molding to be used successfully in the future. Some of the most important of these aspects are listed below:

a. Compatibility between molten nominal 60-Nitinol alloy and a graphite-coated shell mold.

(1) Phenol-formaldehyde resin decomposition promoting alloy-resin reactions

(2) Mold outgassing, partly promoted by the furnace chamber vacuum.

b. Dimensional accuracy of casting.

c. Casting surface finish.

d. Shrinkage porosity of casting, and ways of minimizing.

e. Fluidity of nominal 60-Nitinol.

8. After analyzing the required critical parameters, it became very apparent that the quickest results would be obtained through actually casting 60-Nitinol in a standard shell mold. To implement this decision, representative shell molds were provided by the Lynchburg Foundry (Lynchburg, Virginia). Because of the limited capacity of the vacuum melting furnace, the mold used by Lynchburg Foundry to make "doughnut" shaped flat discs was used. A half section of this shell mold is shown (without core) in Figure 1.

9. The melting of 60-Nitinol, because of the reactivity of the molten alloy with air, must be performed in vacuum or an inert controlled atmosphere. For this part of the experiment a Stokes vacuum

induction melting furnace was used. The graphite crucible of this furnace will hold about 12 to 14 pounds maximum of the 60-Nitinol. In order to save time in alloy preparation, some hot extruded 60-Nitinol scrap bar stock was remelted. The pouring and casting in the shell molding was performed within the furnace vacuum chamber.

10. To perform this experiment two key, but separate, pourings were required. The steps involved in each operation were essentially similar. These general steps were as follows:

a. Graphite coat shell mold surface with a very thin layer of Aqua Dag, which is a suspension of fine graphite particles in a water solution.

b. Place coated mold in an oven heated above the boiling point of water.

c. Charge the prealloyed 60-Nitinol to the graphite melting crucible.

d. Place heated and dried shell mold in a container with suitable metal shot to provide uniform support (and heat transfer) for their shells.

e. Place mold assembly into the furnace chamber in position to receive molten 60-Nitinol.

f. Close chamber and evacuate.

g. Initiate power flow through induction coils.

h. Monitor alloy melting and adjust melt temperature optically through sight glass.

i. Pour temperature-adjusted melt (corrected to about 1450 to 1500°C) into shell mold.

j. Stop power flow through induction coil.

k. After the 60-Nitinol has solidified and cooled sufficiently the furnace chamber is opened.

l. The charred molding is removed from the casting by mechanical chipping followed by some form of grit blasting. Figures 1 and 2 show the results of pouring into an open half mold.

m. The resultant castings were thoroughly analyzed and the results of those analyses are included in the following section.

EXPERIMENTAL RESULTS

60-Nitinol/Shell Molding Compatibility

11. Initial observations of possible molten alloy-shell mold gassing were made during the actual pouring. The melting furnace operator observed little, if any, sign of outgassing. Postcasting analysis, which consisted mainly of metallography and microhardness measurements, tended to confirm initial observations. Figure 3 shows a photomicrograph (250 magnifications) of the shell mold cast 60-Nitinol taken near the mold-alloy interface. In a general sense the photomicrograph shows some excess $TiNi_3$ phase and the normal Ti_4Ni_2O and comparable nitride contaminants. However, there is no strong visual evidence of carbide phase (e.g., TiC) as a result of a reaction between the organic resin of the shell mold and the 60-Nitinol. Further, microhardness measurements were made in both the inner bulk portion of the casting and the edge near the casting surface. No measurable differences were observed.

12. In the initial casting of half-discs in the half-mold section the major concern was compatibility. No provision was made in this exercise to minimize casting porosity. However, when the sectioned casting did show considerable porosity of varying size, concern was directed to the origin of the porosity. Was it normal shrinkage porosity or was it gaseous mold decomposition products? The two photomicrographs shown in Figure 4 tend to indicate normal shrinkage porosity associated with the lack of proper molten feed alloy. This early conclusion was confirmed when a second full-disc casting was made. The whole and sectioned discs are shown in Figures 5 and 6. The latter experimental casting, while still inadequate from a molten feed standpoint, confined most of its porosity to one large void at the center top. An X-ray radiograph, shown in Figure 7, also shows the shrinkage void. The void position appears displaced because of the casting orientation during radiography.

13. In summary, there appears to be reasonably good compatibility between molten 60-Nitinol and a properly coated shell mold.

Dimensional Accuracy and Surface Finish

14. In order to attain reasonable dimensional accuracy in cast components, compensation must be made for such things as thermally-induced mold wall movement, solidification shrinkage and thermal contraction of solidified castings. Since hand tools generally have relatively low weight and small cross-sectional thickness the factor most apt to affect casting accuracy is solidification shrinkage.

15. Table II, in a very cursory way, summarizes some of the liquid-to-solid shrinkage experienced in the cast 60-Nitinol. The 60-Nitinol experiences a shrinkage (in shell molding) which is roughly 2 to 3 times that of gray cast iron ($\sim$ 1%)[5] and equivalent to or

larger than cast steel[5] (∿ 2%). While the solidification shrinkage of 60-Nitinol is significant it would appear that it can be compensated for through proper pattern design and allowance.

16. No quantitative measurements were made to precisely determine the surface finish. However, it has been reported to vary between 125 and 250 micro-inch for standard shell molding practice (see Appendix A). The authors were advised[6] that surface finish was a function of the molding sand fineness and particle size distribution. Better finishes than those attained in this experiment should be possible using other sand mixes. A qualitative indication of the surface finish attained, using standard shell molding, is shown in Figure 5. It should be further noted that the identification numbers, given on the original pattern, are very clearly and precisely reproduced.

Shrinkage Porosity

17. Table II provides some preliminary information on the extent of liquid-to-solid shrinkage occurring in 60-Nitinol. This shrinkage, while it is somewhat higher than gray cast iron,[5] is still of a magnitude that probably can be handled through pattern compensation. Of more concern is the possible shrinkage porosity and piping that may result in faulty tool castings. Figures 2, 4, 6 and 7 illustrate the potential problem. The casting shown in Figures 5, 6 and 7 is far better than the initial "surface compatibility" casting study shown in Figure 2.

18. However, in neither case was the casting experiment performed with shrinkage porosity solely in mind. As a result microporosity and macroporosity are very prevalent in the initial casting effort and piping is very evident in the second casting. With the relatively larger liquid metal-to-solid metal shrinkage present in 60-Nitinol more care will have to be given to solidification mechanics. For example, larger molten feeder reservoirs will be needed, mold backup material (see Appendix A) may be required to be selectively placed in order to promote gradient heat flow causing a more orderly directed solidification, etc. These and other schemes should provide essentially sound 60-Nitinol tool castings.

Fluidity of Molten 60-Nitinol

19. The term "fluidity" refers to the property of a metal which allows it to flow freely and evenly into a mold and fill it before such freezing occurs as would offer an obstruction to its further flow.[7] Some key factors affecting fluidity are surface tension, surface oxide films, gas content, suspended inclusions, form of crystallization, etc. In the present study no effort was made to carefully measure "fluidity" or the "coefficient of liquidity" of 60-Nitinol.

20. As an adjunct matter some qualitative indications of fluidity were observed. This may be best seen in Figures 5 and 6. Here, in addition to the normal mold part line fins, a large well defined fin was formed adjacent to a graphite "blocking plate." The latter fin is shown at the bottom of Figures 5 and 6. Based upon this cursory evidence and the precise manner in which surface definition was attained (see numbers in Fig. 5) it has been concluded that nominal 60-Nitinol possesses adequate casting fluidity.

Thermal-Metallurgical Response of Cast 60-Nitinol

21. A very important consideration in the ultimate suitability of cast 60-Nitinol tools is their mechanical properties. What effect, if any, does shell mold casting have on the mechanical properties? To provide some insight in this area, hardness of variously thermally treated 60-Nitinol was measured. Direct comparisons were made between the original hot extruded melt stock and the shell mold castings produced from that stock. Table III summarizes and compares average hardness data before and after casting. The hardness given in the first row under "cast hardness" cannot be compared because of vast possible variance due to cooling rate. The R_C 46-47 values are merely given to shed some light on the mass cooling rate within the casting.

22. A comparison of the "furnace cool" and "quenched" hardness values in Table III is significant. The quench-hardening response is precisely the same and at an expected level. The furnace cooled data shows some variance but appears to be in a proper direction. That is, if the casting process contaminated the 60-Nitinol the hardness should increase--yet it actually dropped. While there is no simple explanation for the hardness variation, it appears safe to assume shell molding does not degrade the cast 60-Nitinol product.

Magnetic Properties of Shell Molded 60-Nitinol

23. Magnetic effects measurements were made on samples from each casting. Some form of grit blasting was used to clean the surfaces of each sample prior to measurement. The magnetic effect measured for all cast samples was less than 0.001 millioersted and is negligible.

CONCLUSIONS

24. In general nominal 60-Nitinol alloy appears to be quite compatible with standard shell molding that has been graphite coated. Further, any "breaking down" of the graphite coating if it occurred was not detrimental to the cast product. Such a "washing" or "break down" of the graphite film if it does occur apparently causes the decomposition of the phenol-formaldehyde into carbonaceous products which are compatible with the TiNi-base 60-Nitinol material.[6]

25. Based upon this preliminary study it appears that most concern will be focused upon shrinkage problems, both as it affects dimensional accuracy and shrinkage porosity within the cast part.

FUTURE WORK

26. It is the plan of the present investigators to shell mold cast some representative EOD tools. Suitable steel patterns will be prepared, powdered sand-resin mixtures will be acquired and shell molds will be made. All of these operations will be performed at the U. S. Naval Ordnance Laboratory. Following the preparation of suitable shell molds, casting will be performed in the in-house vacuum induction melting furnace. Thorough analysis of the resulting castings should shed light on the problem areas, costs, production steps, etc. Cast tools will be evaluated in house according to ASTM standards and further tested by the EOD Facility for usability. A report summarizing this latter work should provide a basic guide to future cast 60-Nitinol EOD tool procurement and production.

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The authors wish to express their appreciation for the very helpful discussions and assistance provided by Mr. Larson Wile of the Lynchburg Foundry, Lynchburg, Virginia.

TABLE I

COMPARISON OF "AS CAST" AND "HOT WROUGHT" 60-NITINOL TENSILE AND HARDNESS PROPERTIES

Alloy Preparation	Mechanical Property				
	Ultimate Tensile Strength (Ksi)	Yield Strength (Ksi)	Elongation (%)	Young's Modulus (psi x 10-6)	Average Hardness (R_c)
<u>Wrought Alloy</u>					
Hot extruded	178	*	< 1	15.3	51
Quenched	154	*	< 1	16.5	60
Furnace Cooled	137	51	7	14.2	35
<u>Arc-Cast Alloy</u>					
As Cast	80-140†	*	< 1	14	54
Quenched	120-140	*	< 1	14.5	60
Furnace Cooled	80-95	23-25	< 1	10.3	29

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* Yield strength not precisely measured, but approaches the value for ultimate tensile strength.

† Strength and other properties vary with cooling rate of casting.

TABLE II

DATA ON THE SHRINKAGE OF NOMINAL 60-NITINOL WHEN CAST IN A SHELL MOLD

Position of Measurement*	Mold Dimension (in)	Cast Part Dimension (in)	Shrinkage (%)**
Width of disc where core intersects disc	0.672±.003	0.660±.004	1.78
Width at outer edge of disc	0.659±.003	0.640±.004	2.89
Diameter of disc	3.970±.005	3.898±.006	1.81

---

* Subject part was a circular disc with a hole (1-5/8") in the center.

** % shrinkage was calculated from the <u>mean</u> dimensions, using the following equation:

$$\frac{\text{Dimension (mold)} - \text{Dimension (cast part)}}{\text{Dimension (mold)}} \times 100$$

TABLE III

COMPARISON OF AVERAGE HARDNESS DATA TAKEN FROM THE ORIGINAL 60-NITINOL MELTING STOCK (EXTRUDED) AND THE SHELL MOLDED CAST PRODUCT

	Average Hardness (R_C)	
Condition	Original Melting Stock (extruded)	After Remelting and Casting
Cast Hardness-- normal cooling rate	--	46-47
Thermal treatment-- 950°C, 1 hour, furnace cool	35	29
Thermal treatment-- 950°C, 1 hour, water quench	60	60

REFERENCES

1. "60-Nitinol Alloys," brochure prepared at the U. S. Naval Ordnance Laboratory and printed by the Government Printing Office, No. 931-002, April 1967.

2. Unpublished data residing in U. S. Naval Ordnance Laboratory files.

3. W. J. Buehler, U. S. Patents: 3,529,958; 3,672,879 and 3,679,394.

4. Internal memorandum.

5. Excerpts from Metals Handbook, Vol. 5, entitled "Forging and Casting," American Society for Metals, 1970.

6. Private communication with Mr. Larson Wiles at the Lynchburg Foundry, Lynchburg, Va.

7. Metals Handbook, 1948 Edition, American Society for Metals, p 199, 1948.

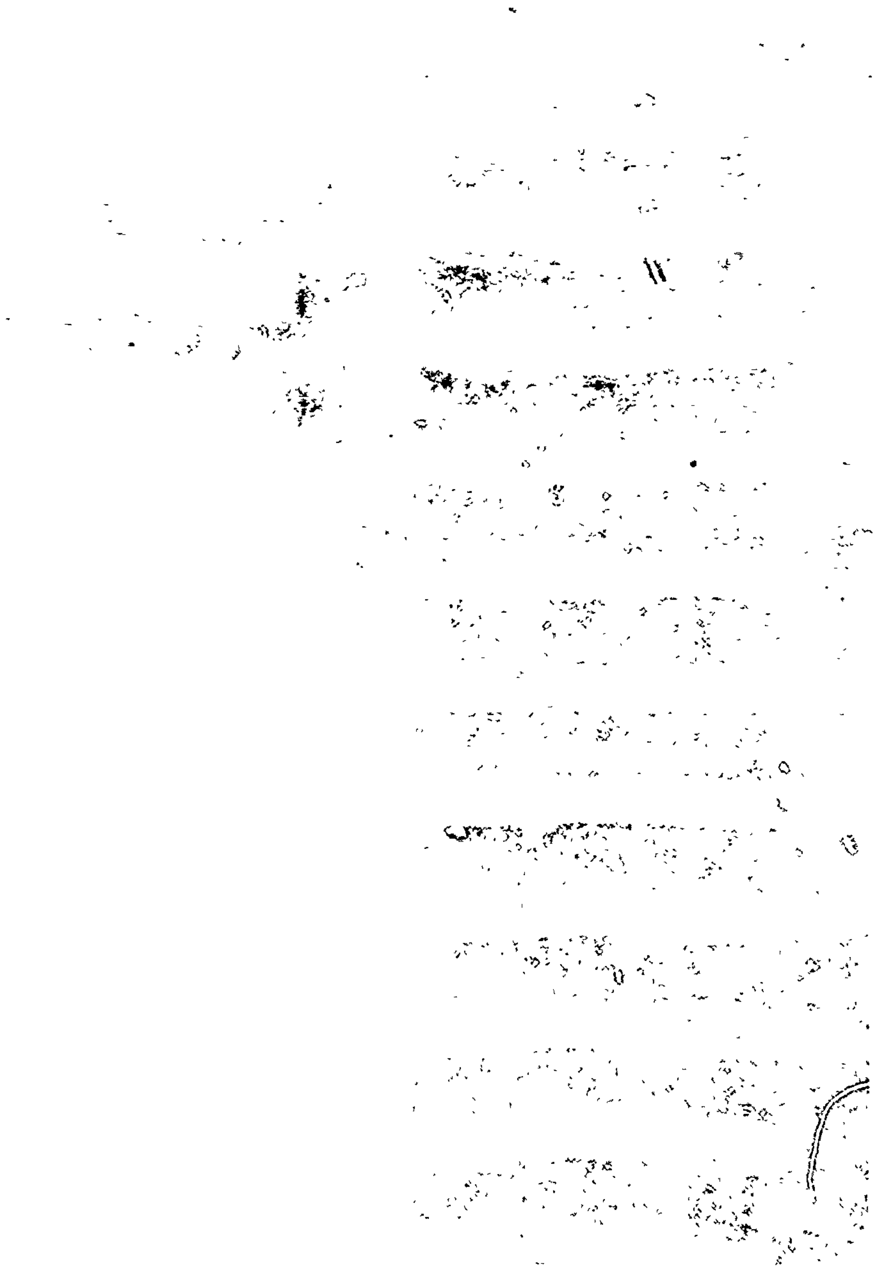

Fig. 1. Half section of shell mold is shown at right of photograph. At left are three half-discs produced by pouring directly into the open shell mold. Study was performed to determine the compatibility between 60-Nitinol and graphite-coated shell molding.

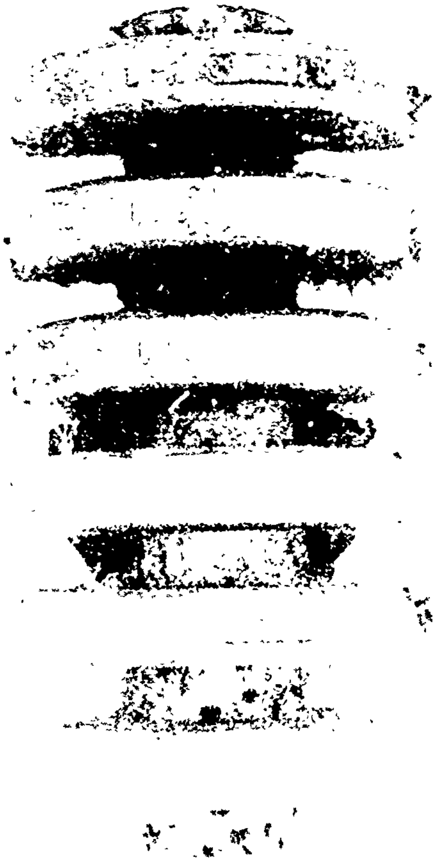

Fig. 2. Results of forming casting by pouring into graphite-coated half mold. Note porosity resulting from lack of a suitable molten alloy reservoir.

Fig. 3. Photomicrograph of cast 60-Nitinol produced in shell molding. Section shown was taken near mold-metal interface. Note minimal evidence of 60-Nitinol contamination. 250 magnifications. Etchout: HF, HNO_3 and H_2O.

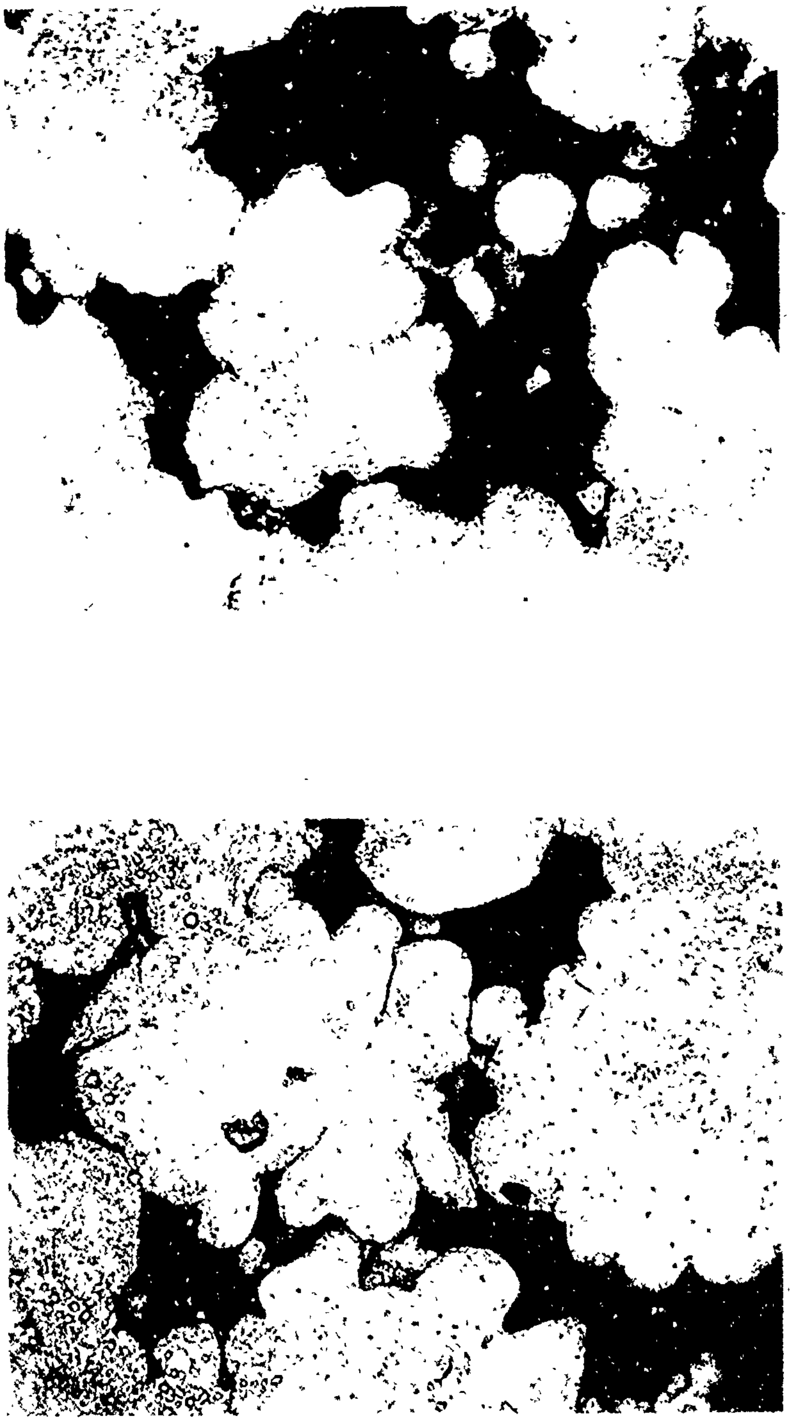

Fig. 4. Two views of porous portion of 60-Nitinol half-disc castings. Rounded void area indicates shrinkage porosity. 250 magnifications.

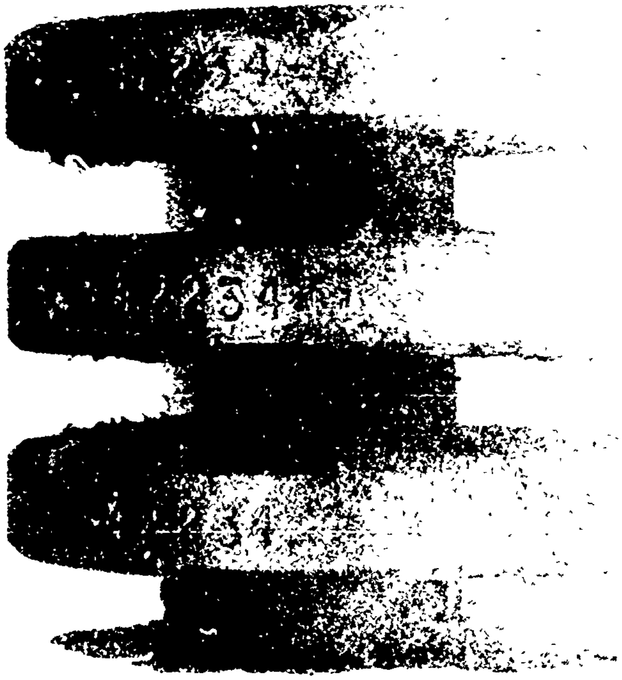

Fig. 5. Photograph of three cast 60-Nitinol discs produced by using graphite-coated shell molding.

Fig. 6. Cast 60-Nitinol discs diametrically sectioned. Note "flash" of cast metal produced at part line and shrinkage void caused by limited supply of molten alloy.

Fig. 7. X-ray radiograph of cast whole 60-Nitinol discs. Note shrinkage void (arrow) caused by limited available feed metal.

APPENDIX A

SHELL MOLDING is a process in which a mold is formed from a mixture of sand and a thermosetting resin binder that is placed against a heated metal pattern. When the mixture is heated in this manner, the resin cures, causing the sand grains to adhere to each other, forming a sturdy shell that constitutes half of a mold. After the shell has been cured and stripped from the pattern, any cores required are set, the cope and drag halves of the mold are secured together, placed in a flask and backup material added; then the mold is ready to be poured.

The shell mold process is particularly suited to castings for which:

1. The greater dimensional accuracy attainable with shell molding (as compared with conventional green sand molding) can reduce the amount of machining required for completion of the part.

2. As-cast dimensions may not be critical, but smooth surfaces (smoother than can be obtained in green sand) are the primary objective.

In addition to producing castings that have greater accuracy and smoother surfaces, shell molding has two other advantages over green sand molding: (a) less sand required, and (b) fewer restrictions on casting design.

The limitations or disadvantages of shell mold casting are:

1. Maximum casting size and weight are limited (see above).

2. High cost of patterns, which must be machined from metal.

3. High cost of resin binder.

4. Relative inflexibility in gating and risering. Gates and risers must be incorporated, at least in part, into the shell mold pattern.

5. Shrinkage factors vary with casting practice. (Two foundries using the same pattern may pour castings with different dimensional variations.)

6. More equipment and control facilities are needed, such as for heated metal patterns.

Mold Backup. To obtain accurate dimensional control of castings made in shell molds, especially those of large size and thick sections, the relatively thin molds usually must be supported over the entire outer surface while the casting is being poured. Support permits the use of molds with thinner walls, thus reducing shell costs. Backup also helps to prevent runouts and casting bleeders, and provides a suitable bed for mold positioning. In the casting of malleable iron, backup material may be required for control of the cooling rate of castings to prevent mottling or primary graphitization during solidification.

Backup Material. Various means and materials have been used to support or back up shell molds during casting and the initial cooling period.

To be successful, the backup means or material must rigidly support the shell mold against the pressure of the molten metal in the internal cavity during the casting period. It must not, however, exert a pressure on the outside of the shell mold in excess of the internal pressure, as this would cause the mold wall to move toward the molten metal and would result in casting distortion just as serious as an outward movement.

The most feasible backup of shell molds for a large variety of high-production castings is a granular material having semifluid characteristics and a density approaching that of the casting being poured. A choice between the two common backup materials (shot and gravel) depends mainly on consideration of the more effective cooling of shot against the lower cost of gravel.

Cast iron shot is generally preferred for backup on high-production, mechanized shell molding lines. It is readily available, will flow when vibrated because of the spherical shape of the particles in a manner similar to a true fluid, and will pack to maximum density around the shell molds. Even though shot approaches true fluid characteristics while being vibrated, it must not produce pressure on the outside of the shell mold in excess of the internal pressure during pouring.

The fluid nature of the shot under vibration accomplishes a uniform support over all areas of the outer surface of the shell molds, especially beneath horizontal projections. This characteristic must be maintained as high and as nearly constant as possible for uniform control of casting dimensions.

Backup shot is controlled by a specification of shot diameter and weight per cubic foot. Controlling the shape and soundness of the shot results in optimum fluidity and packing characteristics.

Metal-Mold Reactions. Because of the relatively high binder content in shell molds and cores, mold gases are produced that consist primarily of hydrocarbons, hydrogen, and carbon monoxide. Varying amounts of water vapor and carbon dioxide will also be present, depending on the availability of oxygen in the mold.

The products of shell-binder decomposition have little or no effect on the surface quality of many cast irons and medium-carbon or high-carbon steels. However, low-carbon ferrous materials and alloys that contain strong carbide formers (stainless steels, for example) are subject to gas-induced surface defects and carburizing action when cast in shell molds. In plain-carbon and low-alloy steel castings, the gas-metal reaction is manifested by surface gas pockets and subsurface pinholing, and by carburization of the surface to a concentration of about 0.30% for a depth of 0.050 to 0.100 in. The surface voids are probably caused by rapid buildup of gas pressure within the mold before casting-skin formation has proceeded sufficiently to withstand penetration or deformation by the gas.

Although some surface carburization of mild steel castings is seldom significant, shell mold casting of alloys containing appreciable amounts of strong carbide formers (chromium, tungsten, molybdenum and vanadium) results in carbide formation at surface grain boundaries. Subsequent heat treatment of such castings may result in surface cracking. Re-solution or spheroidization of the grain-boundary carbides is not feasible, because of the high temperatures and long heating times required.

The mold-metal reaction and carbon pickup in shell mold casting can be minimized by: (a) reducing the mold gas available at the metal-mold interface, (b) increasing the rate at which the casting skin solidifies, or (c) chemically modifying the mold material and the casting metal.

Dimensional Accuracy. Castings made in shell molds are generally more accurate dimensionally than sand castings, and they can be held to closer tolerances. Problems concerning dimensional accuracy do arise, however, in the shell mold casting process, and many of these are similar to dimensional problems encountered in sand casting.

Surface Finish. Shell molds impart a smoother surface to castings than molds made from green sand or baked sand, as shown by the following comparison for small steel castings (up to 5 lb weight) made by three processes:

Shell mold	125 to 250 micro-in.
Baked sand mold	250 to 500
Green sand mold	500 to 1000

Shell Mold Casting vs Alternative Processes. In most applications in which there is an alternative method to shell molding for the production of a particular part, the alternative is another molding method for producing the part as a casting. Some shell mold castings, however, may be producible by other metal-forming processes, such as closed-die forging.

Shell Mold Casting vs Forging. Before shell mold casting can be considered as a replacement for forging, it must first be established that the properties of a casting are acceptable--regardless of any cost advantage or increase in dimensional control offered by shell molding. There are numerous applications in which castings are acceptable and have replaced forgings.

Shell vs Green Sand Casting. For many applications, the shell mold process produces castings that weigh less than comparable green sand mold castings, require less machining, are more accurate, and have a smoother finish.