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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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FURTHER INVESTIGATION OF THE FEASIBILITY OF THE FREEZE-CASTING METHOD FOR FORMING FULL-SIZE INFILTRATED TITANIUM CARBIDE TURBINE BLADES

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SUMMARY

A method of casting full-size cermet turbine blades from titanium carbide was investigated. An extremely thick slip of titanium carbide was prepared with a small amount of binder, cast into a mold by vibration, centrifuged, and frozen to retain the shape of the mold. The casting was then dried in an absorbent sand and sintered by conventional means. The porous casting could then be infiltrated with a nickel- or cobalt-base alloy. Densities and radiographs of the blade prior to infiltration were used as criteria for soundness.

INTRODUCTION

Because of outstanding strength and oxidation resistance at high temperatures, many of the refractory carbides, oxides, silicides, borides, and cermets have been proposed for use as turbine-blade materials. Present methods of forming these materials consist of grinding either fully sintered or semisintered blanks to shape. Because these grinding operations are both time-consuming and costly, methods of eliminating or minimizing them are of interest.

A method for preparing intricately shaped objects from refractory powders without grinding is reported in reference 1. Briefly, that method consists of preparing an extremely thick slip of such a material as titanium carbide with a small amount of binder, casting the slip into a mold, and freezing to retain the shape of the mold. The casting is then dried by sublimation, sintered or semisintered, and infiltrated. To demonstrate the feasibility of that method, small turbine blades (fig. 1(a)) were prepared.

When this work was extended to full-size turbine blades (fig. 1(b)), the following problems were encountered:

(1) Segregations and large holes were found in the casting, particularly in the root area.

(2) In the as-cast state, the castings had excessively low densities.

(3) Gradations in density were found along the lengths of the blades.

The objective of this investigation was to determine whether these difficulties can be overcome and to demonstrate that it is possible to make a full-size porous titanium carbide turbine blade suitable for infiltration. The densities and radiographs of presintered compacts prior to infiltration were used as criteria for soundness.

PROCEDURE

Freeze-Casting Method

The freeze-casting method that was developed is shown in flowsheet form in figure 2. The processes are discussed in this section in the order shown in the figure.

Powder. - The titanium carbide powder was produced by Kennametal Inc. and had an average particle size of 3 to 5 microns. The powder contained no paraffin or binder of any kind.

Preparation of slip. - Titanium carbide slips were prepared by mixing 220 grams of titanium carbide powder with 40 milliliters of a binder consisting of either a 40 percent by weight aqueous suspension of latex or a 2.5 percent aqueous suspension of cornstarch.

Although both types of binders were successfully used, the latex suspension resulted in castings which dried more readily and had better green strength and, consequently, was used for most of the castings. The specimens containing this binder also had the additional advantage that they hardened more than those containing starch during the centrifuging operation. All slips were mixed by vibrating the mixture of powder and liquid with a constant frequency (60 cycles) and a variable-amplitude vibrator. The power input and related amplitude could be adjusted to give rapid mixing. Slips were mixed, set for 2 hours while covered to prevent evaporation of the water, remixed, and cast. In preliminary tests, it was determined that the minimum binder content which would possess good die filling properties was one containing approximately 16 percent binder by volume. Consequently, this ratio of binder to titanium carbide was used throughout this investigation.

Mold. - When considering a mold material for the freeze-cast process, the following requirements are very important:

(1) The frozen binder should not adhere to the mold cavity.

(2) The mold should be light so that it may be readily vibrated with the desired amplitude during the filling operation.

(3) The mold should have sufficient strength to resist the pressures exerted upon it during centrifuging.

(4) The mold material should be easily shaped.

In this investigation a coating was used to prevent the adherence of the casting to the mold. Considerable care is required in the selection of a coating, since it must adhere tightly to the mold and not flow, buckle, or wrinkle during centrifuging. Teflon coatings had the required properties. Ice does not adhere readily to Teflon (0.005-percent water absorption (ref. 2)), and it can be coated onto materials such as aluminum, magnesium, and steel. Because of ease of machining and very low density, magnesium was used as the mold material. Teflon coatings were sintered onto the dies (fig. 3).

As reported in reference 2, Teflon has low density (2.1 to 2.2 g/cc), some strength (1500- to 3000-psi tensile strength at room temperature), and can be readily molded or machined to shape. These properties indicate that solid Teflon might be used for molds for the freeze-cast process; however, this was not investigated.

Casting. - Although the slips were so dry that they would stand without slumping, they were very fluid when vibrated and would flow easily into the mold. The apparatus for casting the slip into the mold is shown in figure 4. Both the mold and hopper containing the slip were vibrated at 60 cycles per minute throughout the casting process; however, the amplitude of each was adjusted for rapid filling. It took about 15 minutes to fill the mold. The slip flowed through the blade base. Initially, while filling the airfoil section, the mold was tilted at a slight angle toward the hopper. When the blade platform was reached, the mold was rotated back to a vertical position to eliminate air pockets in the blade. After the mold was filled, it was vibrated at low amplitude for about 1 hour. During this time approximately 3/4 to 1 inch of liquid came to the top of the mold. This liquid was decanted and discarded.

Centrifuging. - Centrifuging was used to increase and equalize the density of the casting. After the mold was filled, it was placed in the centrifuge (shown in fig. 5) with the blade base toward the center and at a radius of $9\frac{1}{2}$ inches, and counterbalanced. The cycle used for each specimen is shown in the following table:

Spin number	Spinning speed, rpm	Time at spinning speed, min	Amount of liquid decanted after run, ml
1	1500	2	6
2	1900	1/2	0.5
3	1500	2	.5
[a]4	1500	2	.7

[a] A 120-gram lead weight was placed in the mold at the blade base during this spin.

The compact was lightly hand tamped between each spin cycle. If the liquid were not decanted after each run, the total liquid removed by centrifuging would have been much less. During the fourth spinning run a 120-gram lead weight was placed in the mold so that it rested against the root section of the casting. This increased the centrifugal force at that point.

Freezing. - Before freezing the mold, the edges were sealed and lightly clamped together with cellophane tape, which prevented the mold from popping open as the bolts were removed. The mold assembly was then placed in a bath of kerosene, which was held at 25° to 28° F. Freezing times were from 45 minutes to 1 hour.

Casting removal. - After freezing, the casting was taken from the mold. As mentioned previously, the use of Teflon coatings prevented any sticking, and the blade was easily removed. A photograph of an as-cast surface is shown in figure 6. The wrinkle-free surfaces and the absence of any tearing or pullouts can be noted.

In preliminary tests with coatings which were not sufficiently adherent to the mold wall, it was noted that the coatings moved radially outward during centrifuging and resulted in wrinkled surfaces on the castings.

Drying. - The frozen castings were placed on a bed of the drying agent, fuller's earth, and then covered with additional drying agent (fig. 7). The blades were slowly dried for 2 days at room temperature. The drying agent gently supports the casting and provides for a slow uniform removal of the water. This procedure has been found to minimize distortion.

Sintering. - The blades were sintered for 1 hour at 2500° F in a vacuum (pressure of 1 to 5 microns), while being supported by the graphite die shown in figure 8. The die prevented the blade from warping during sintering and acted as a susceptor in the electrical induction field used to provide the heat.

Infiltration. - One of the sintered porous titanium carbide blades was infiltrated with a high-temperature alloy (S-590). This was done by placing the blade in an inverted position in a graphite die with a piece of infiltrated metal of the desired weight placed on top of the blade. Distortion during infiltration was minimized by making the graphite die used for infiltration smaller than the one used for sintering by the amount of the shrinkage that occurred during sintering. The infiltration was accomplished in a vacuum (1 to 5 microns), and, again, the graphite die acted as a susceptor in the induction field. The temperature was raised until the metal became fluid and was held until the metal was absorbed into the porous blade by gravity and capillary action.

Evaluation of Blade Properties

Density. - The densities of sintered and infiltrated blades were determined by the conventional water-immersion method. The sintered porous blades were immersed in a paraffin bath prior to the density determination in order to seal the surface pores. No change in weight was observed while the porous blades were immersed in water.

Soundness. - Radiographs taken with a 140-kilovolt X-ray machine were used as a criterion of soundness. In most cases both high- and low-density exposures were made to improve contrast.

RESULTS AND DISCUSSION

Cast and Sintered Freeze-Cast Blades

Blades made by the freeze-cast method are listed in table I. A photograph of a dried freeze-cast blade is shown in figure 9(a), and a blade after sintering is shown in figure 9(b). Radiographs showed them to be free of holes, segregations, or cracks. A radiograph of blade 2 (table I) after sintering is shown in figure 10. Rough measurements of shrinkage made from the radiographs showed that the shrinkage was approximately 4 percent in the length and 3 percent in the width. A comparison in table II of the densities of various sections of a sintered blade (blade 3, table I) indicates that the porosity of the airfoil varies from 30 to 33 percent. This is believed to be remarkably uniform for a 4-inch length. However, the porosity in the base was 25.1 percent. This decrease was probably caused by the greater packing force exerted on that section by the lead weight added to the mold during centrifuging. The porosity in the root could probably be increased to a value more desirable for infiltration by reducing the size of the lead weight.

Because the centrifugal force exerted near the base was a minimum, it was thought that the force was not sufficient to provide the required packing. Thus, the lead weight had been added to provide additional load on the root. Since this was a feasibility study, no attempt was made to vary the amount of lead and determine the optimum weight.

Infiltrated Freeze-Cast Blade

A freeze-cast blade infiltrated with S-590 alloy is shown in figures 1(a) and 9(c). The hardness and density values for various sections of the infiltrated blade (blade 2, table I) are listed in table III. Both the density and the hardness of the blade are uniform over the entire length. Weights of the blade before and after infiltration indicate that the metal content is approximately 49.5 percent by weight. The microstructure of the infiltrated blade at various sections is shown in figure 11. Examination of the structures shows the increasing separation of the carbide grains as the base of the blade (point of infiltration) is approached. At the top of the airfoil (fig. 11(a)) there is a large amount of interconnected carbides (darker phase). At the center of the airfoil the amount of interconnected carbides has decreased (fig. 11(b)). There is very little of the interconnected carbides at the bottom of the airfoil (fig. 11(c)) and a fairly uniform dispersion of carbide in the base (fig. 11(d)). The amount of porosity decreases as the base is approached. The top of the airfoil (fig. 11(a)) shows a slight amount of porosity (black areas). As the center of the airfoil is approached, the amount of porosity has decreased considerably (fig. 11(b)). At the bottom of the airfoil and in the root section no porosity is visible (figs. 11(c) and (d)). An increase in the carbide grain size near the base of the blade is evident by comparing figures 11(a) and (b) with figures 11(c) and (d). In each case the carbides tend to be rounded rather than angular.

The root section has the longest time in contact with the infiltrated metal during infiltration and is also subject to the greatest degree of solution, since all of the infiltrated metal must pass through this area. The rounding of the carbide in the root microstructure appears to be a result of this increased solution. Heat treatments subsequent to infiltration, holding the blade at temperature after infiltration, or infiltrating over a broader area may result in a more uniform structure throughout an infiltrated blade.

Applicability of Method

Infiltration of a porous titanium carbide turbine blade produced by the freeze-cast method offers a convenient method of producing titanium carbide cermet turbine blades. A large amount of powder waste and machining operations is eliminated. The basic steps involved in freeze casting are shown to be applicable to a variety of refractory powders (ref. 1). It is believed that the variation in the procedure used in this study does not alter this applicability, and the freeze-cast method offers a convenient method of producing turbine blades or other objects of complex shape from other refractory materials such as aluminum oxide or molybdenum disilicide, which are difficult to fabricate.

SUMMARY OF RESULTS

An investigation of a method of freeze-casting full-size porous turbine blades from titanium carbide powder has shown the method to be feasible. Basic steps in the procedure are:

(1) Preparation of an extremely thick slip of the powder in a fluid containing a binder

(2) Vibrating the slip into a mold

(3) Centrifuging the casting to achieve a more dense and uniform body

(4) Freezing the slip to retain the shape of the mold

(5) Removal of the remaining liquid by drying the casting in porous sand such as fuller's earth

(6) Sintering the dried casting

The prepared porous titanium carbide turbine blades were of good appearance and free of flaws. While the blades contained some porosity after infiltration, it is believed that the feasibility of the method has been established, and with further development, blades free of flaws can be produced.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, June 18, 1956

REFERENCES

1. Maxwell, W. A., Gurnick, R. S., and Francisco, A. C.: Preliminary Investigation of the "Freeze-Casting" Method for Forming Refractory Powders. NACA RM E53L21, 1954.

2. Anon.: Teflon - Properties - Uses - Processing Techniques. Polychem. Dept., E. I. DuPont De Nemours & Co., Inc., Wilmington (Delaware), 1955.

TABLE I. - DATA FOR FREEZE-CAST BLADES

Blade number	Density, g/ml		Porosity of sintered casting, percent	Radiograph of			Appearance
	Sintered	Infiltrated		Dried casting	Sintered casting	Infiltrated casting	
1	3.26	(a)	34.1	Good	Good	----	Good
2	3.24	6.17	34.5	Good	Good	Good	Good
3	3.38	(a)	31.7	Good	Good	----	Good

[a] Not infiltrated

TABLE II. - DENSITY AND PERCENT POROSITY OF VARIOUS SECTIONS OF SINTERED FREEZE-CAST BLADE

Blade number	Over-all density of blade, g/ml	Over-all porosity of blade, percent	Top of airfoil		Center of airfoil		Bottom of airfoil		Root	
			Density, g/ml	Porosity, percent	Density, g/ml	Porosity, percent	Density, g/ml	Porosity, percent	Density, g/ml	Porosity, percent
3	3.38	31.7	3.46	30.6	3.34	33.0	3.41	31.5	3.73	25.1

TABLE III. - DENSITY AND HARDNESS OF VARIOUS SECTIONS OF INFILTRATED FREEZE-CAST BLADE

Blade number	Over-all density of blade, g/ml	Top of airfoil		Center of airfoil		Bottom of airfoil		Root	
		Density, g/ml	Hardness, Rockwell A-	Density, g/ml	Hardness, Rockwell A-	Density, g/ml	Hardness, Rockwell A-	Density, g/ml	Hardness, Rockwell A-
2	6.17	6.21	86.4	6.01	85.5	6.08	86.1	6.09	85.8

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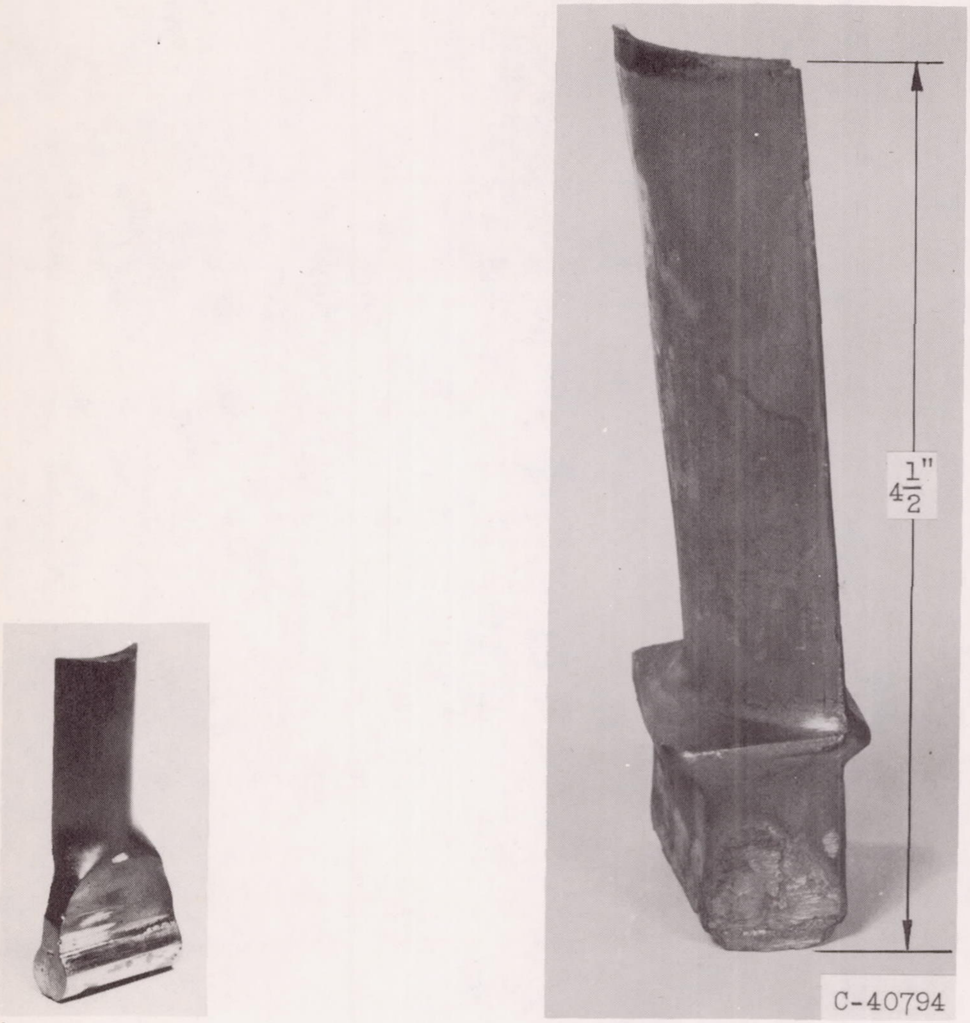

(a) Small size.

(b) Full size.

Figure 1. - Turbine blades prepared from refractory powders without grinding.

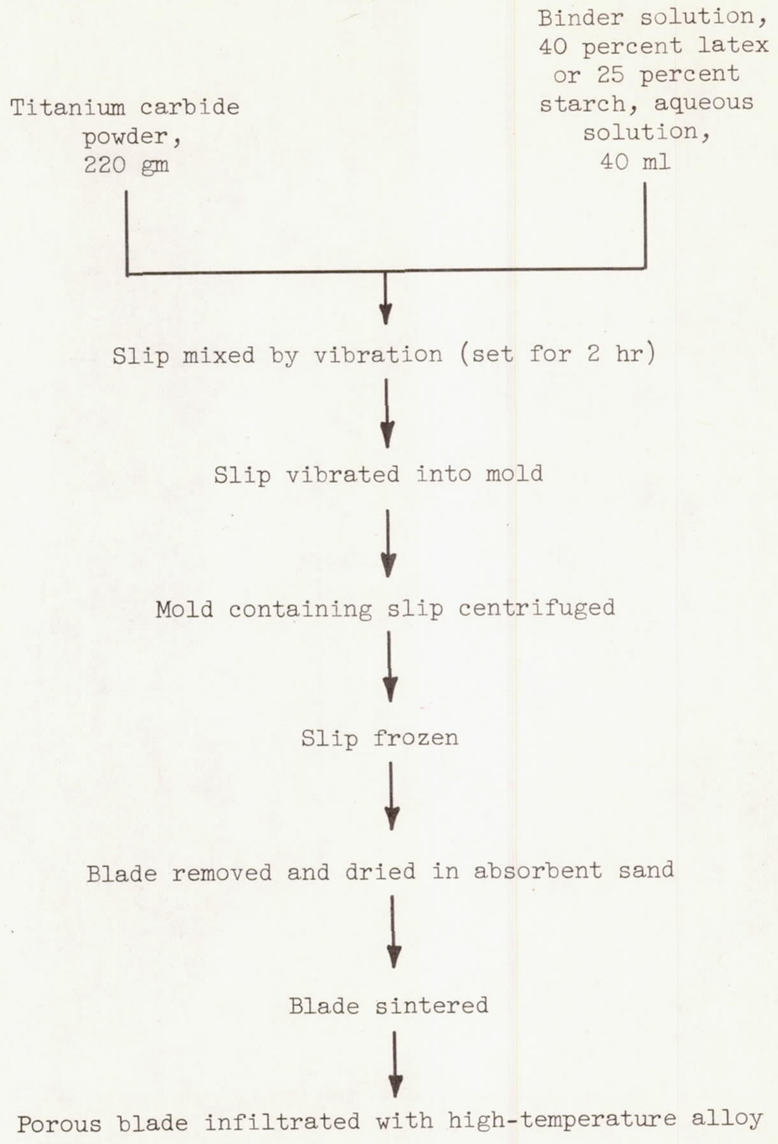

Figure 2. - Flowsheet for preparation of titanium carbide cermet turbine blades.

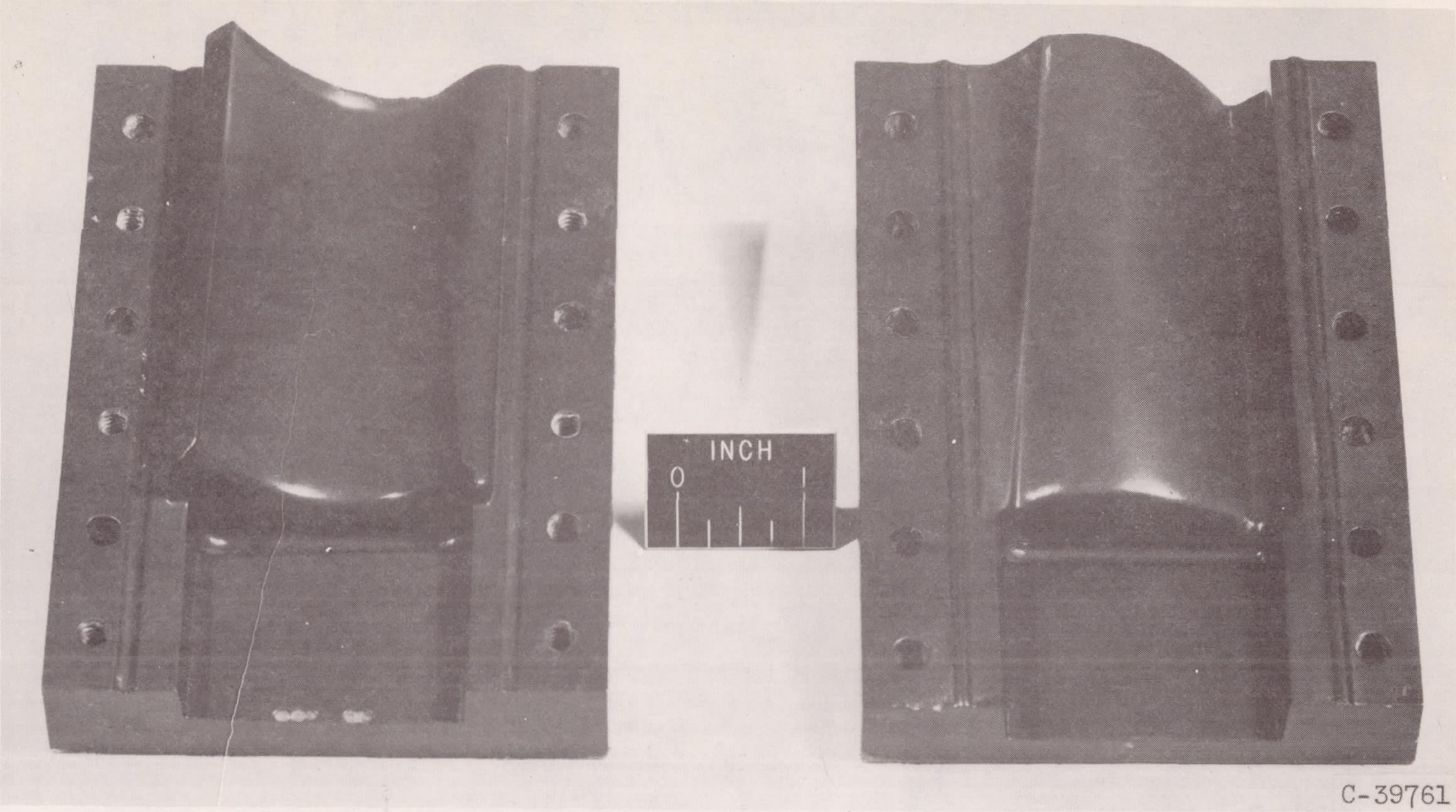

Figure 3. - Magnesium dies coated with Teflon.

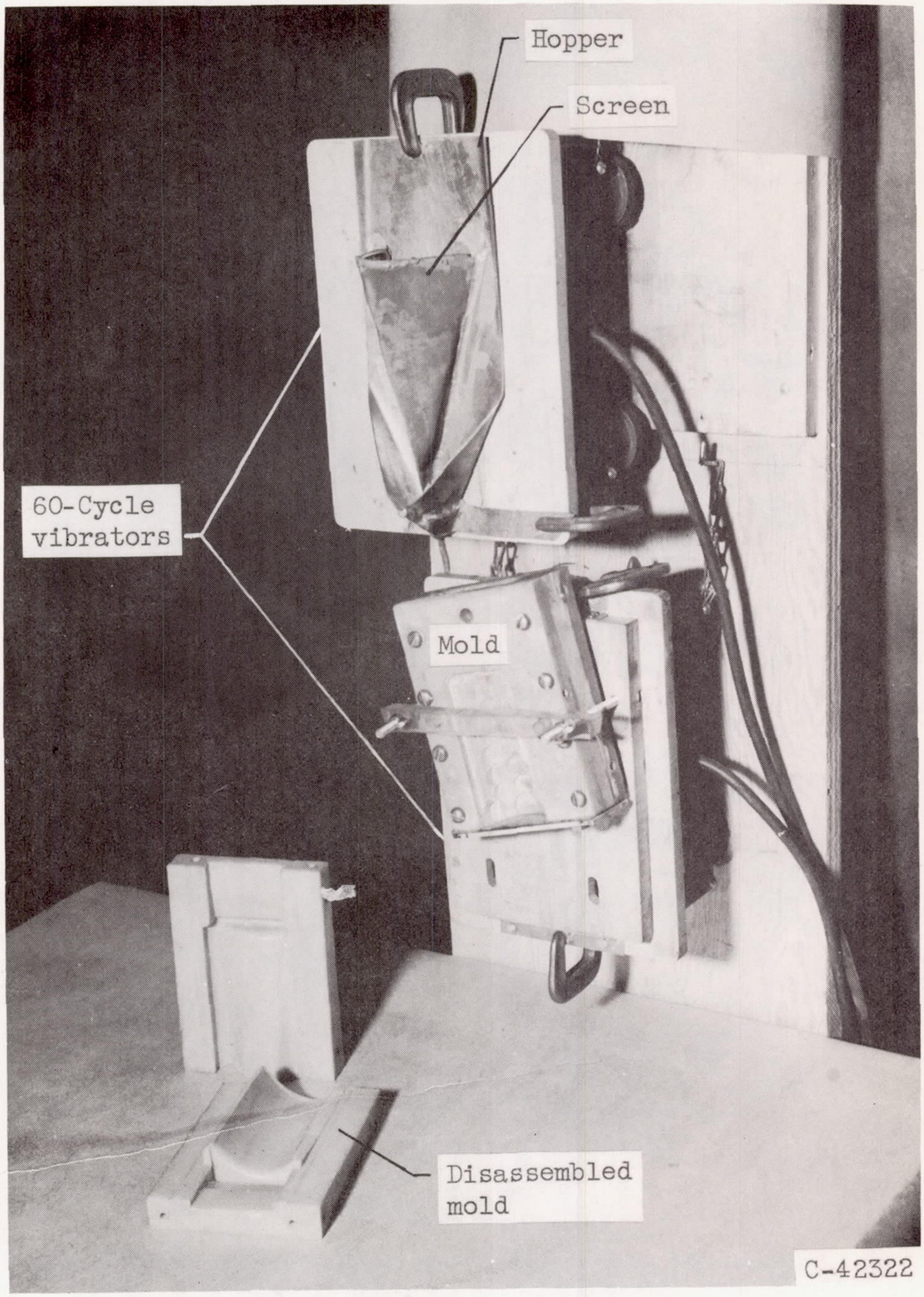

Figure 4. - Vibration-casting unit for freeze-cast process.

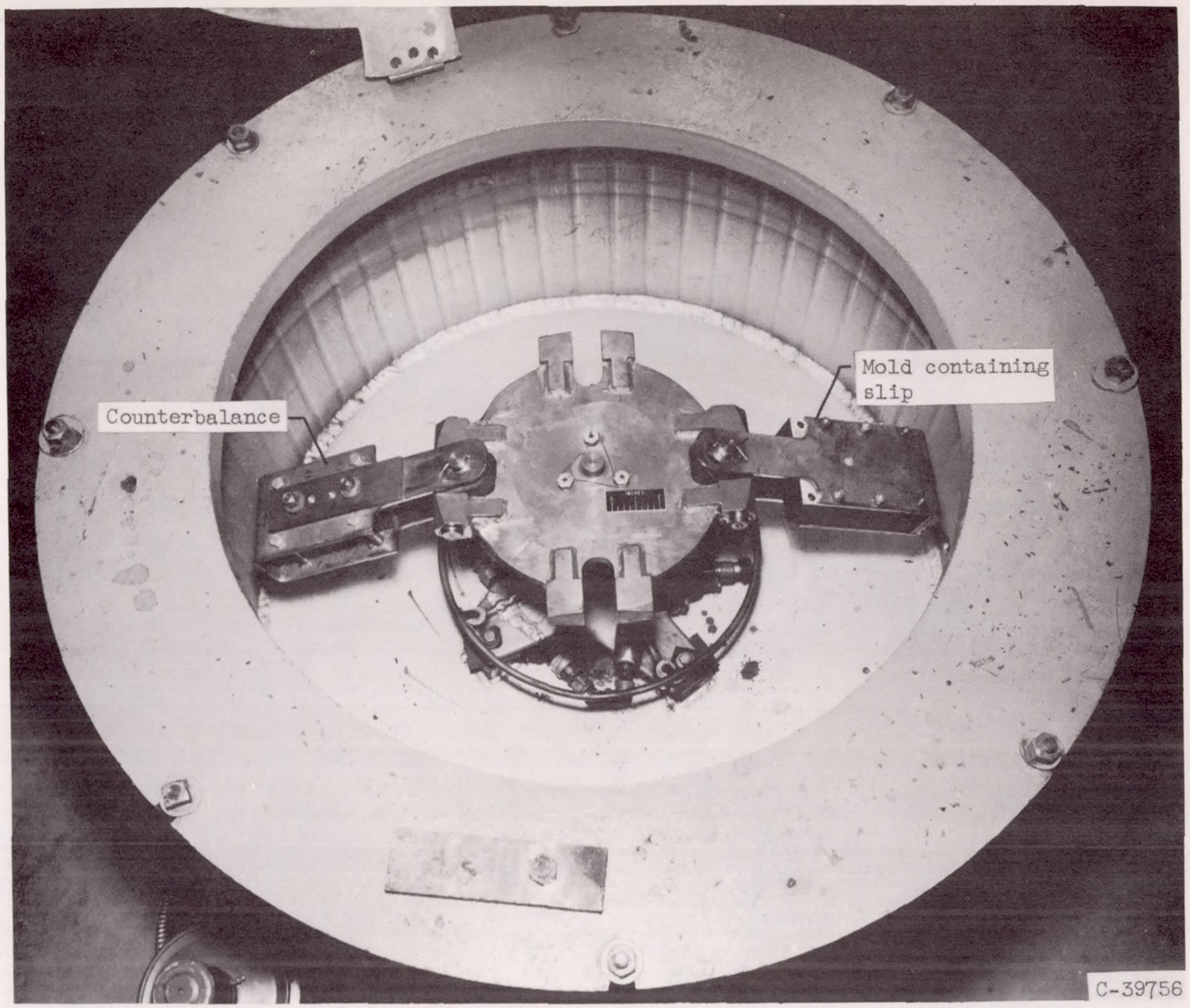

Figure 5. - Centrifuge used for densifying cast powders.

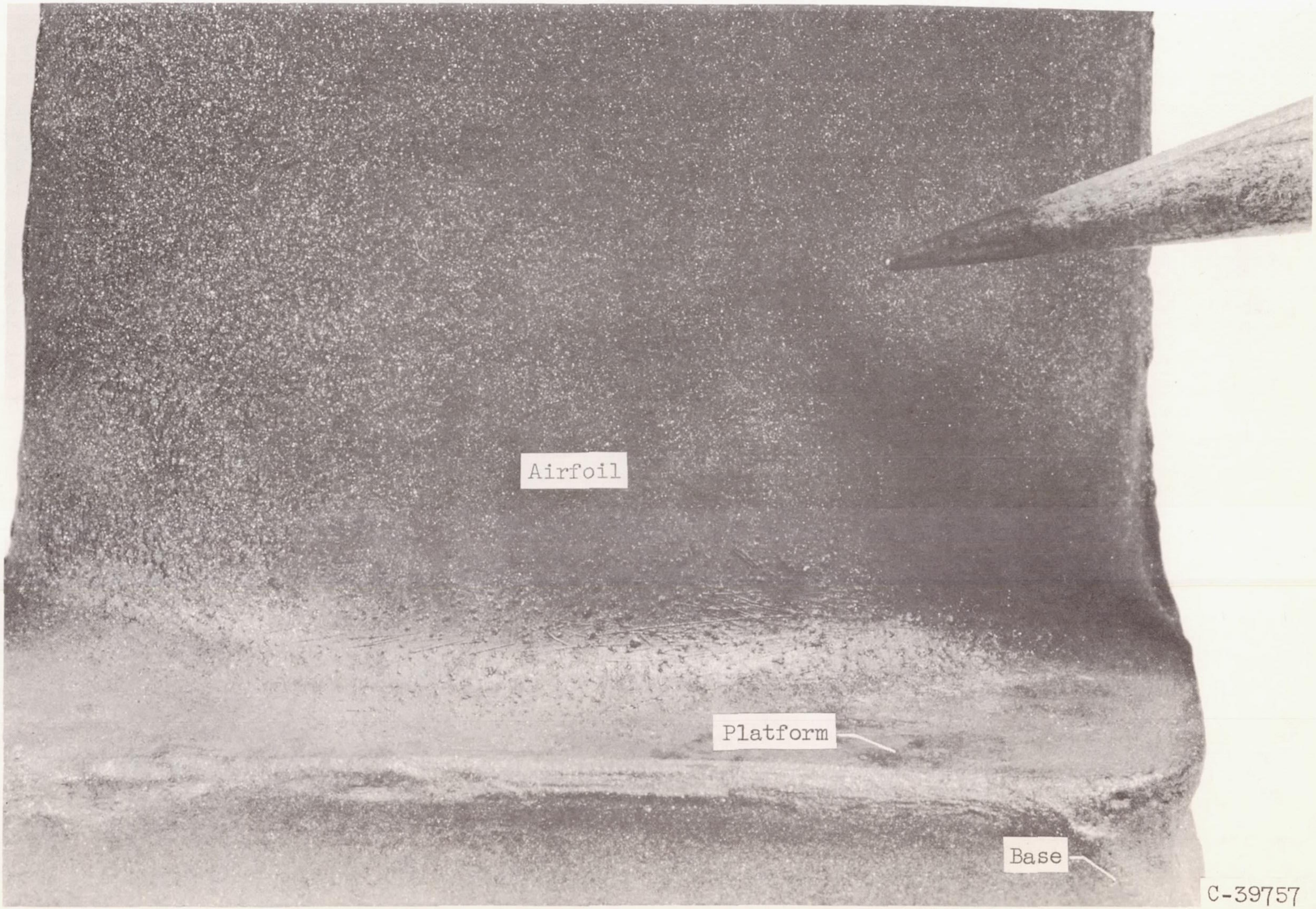

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Figure 6. - Surface of freeze-cast blade as dried (prior to sintering).

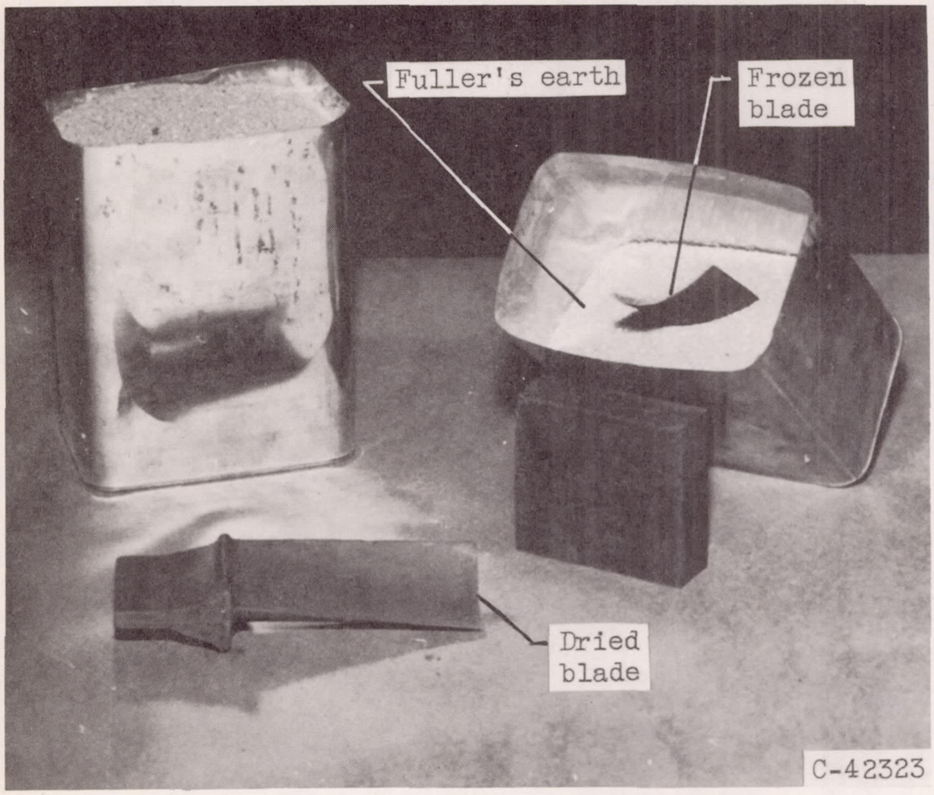


Figure 7. - Drying of frozen freeze-cast blades.

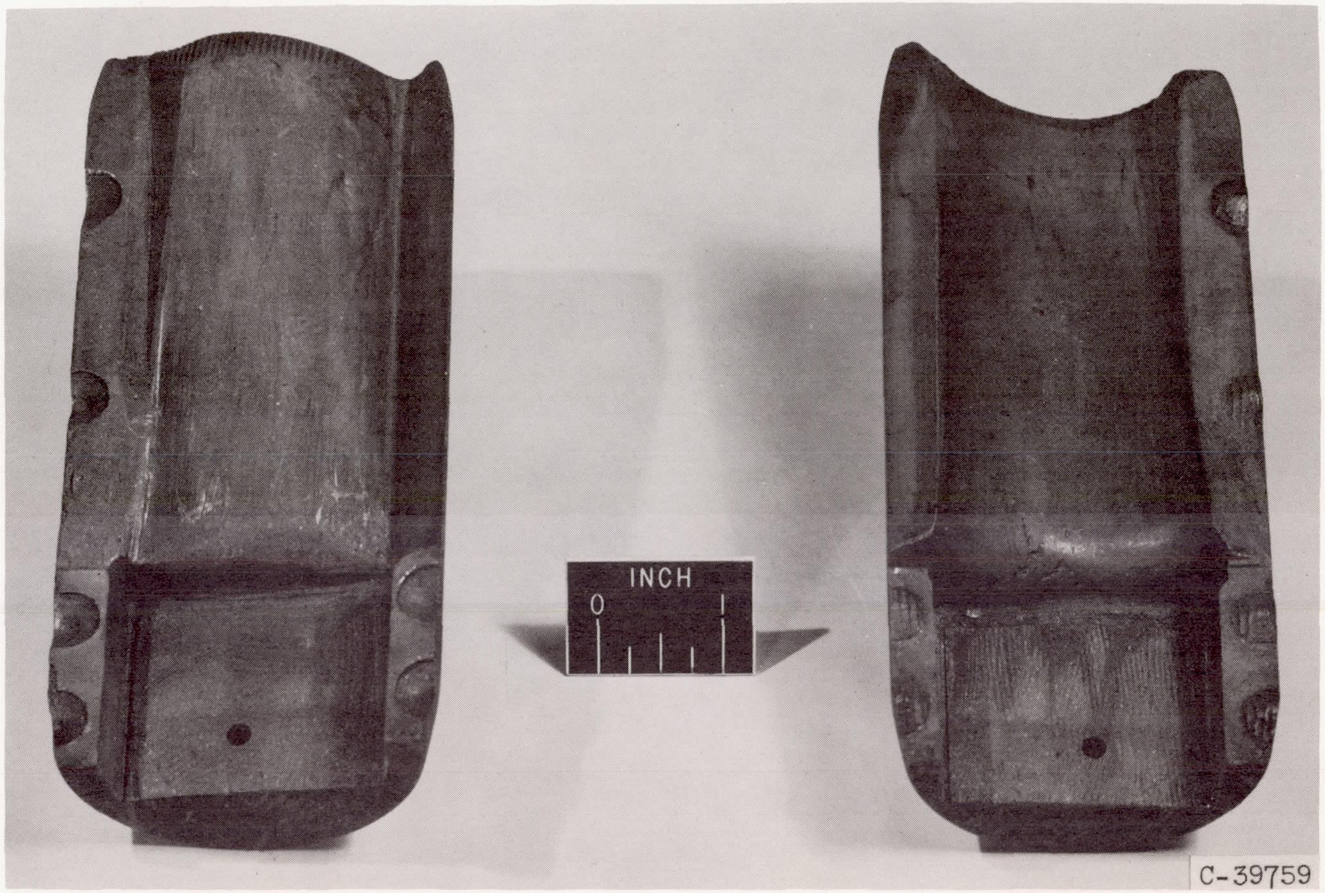
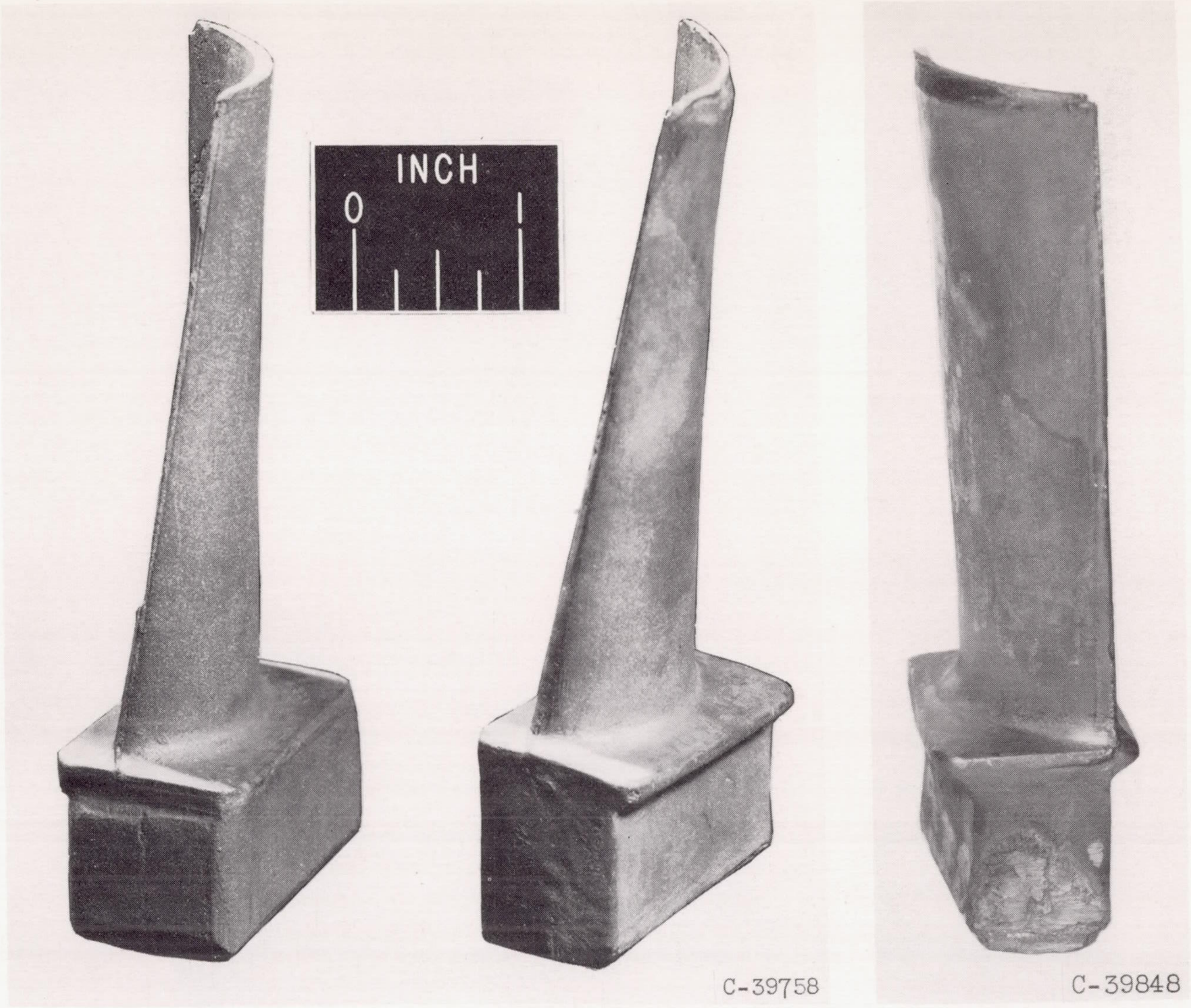


Figure 8. - Graphite die used for supporting freeze-cast blades during sintering.

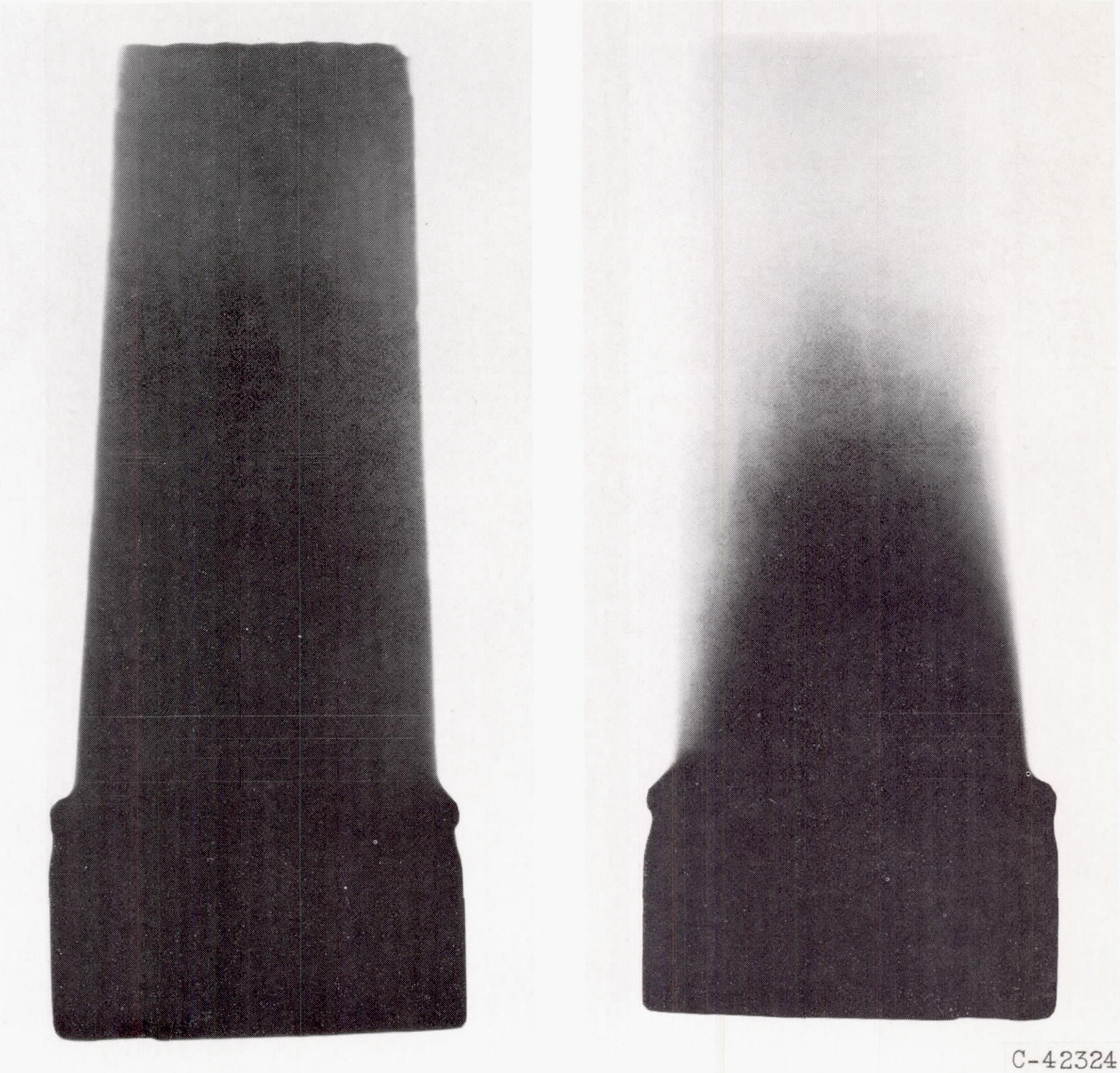


(a) Dried.

(b) Sintered.

(c) Infiltrated.

Figure 9. - Freeze-cast blades at various stages of preparation.



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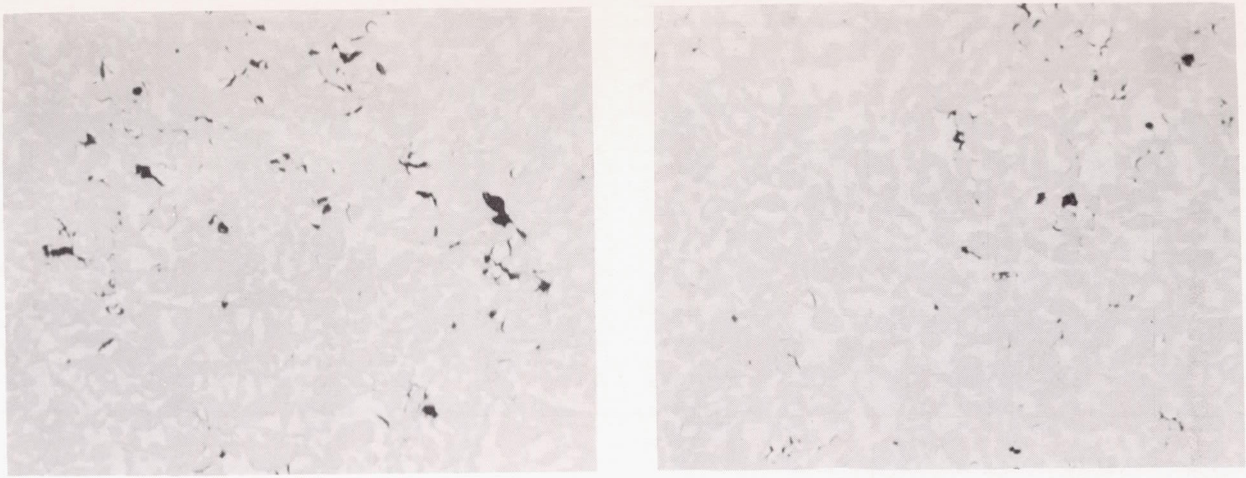
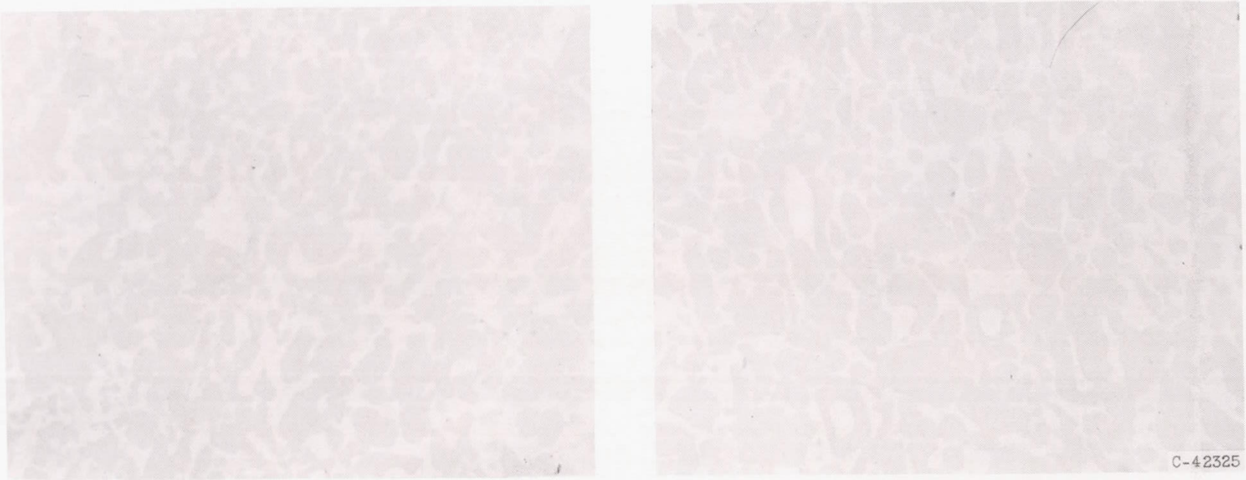
(a) Airfoil. (b) Root.

Figure 10. - Radiograph of sintered titanium carbide blade (blade 2, table I).



(a) Top of airfoil.

(b) Middle of airfoil.



(c) Bottom of airfoil.

(d) Root.

Figure 11. - Microstructure at various sections of titanium carbide freeze-cast blade infiltrated with alloy S-590. No etchant; X1000.

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