Molding of fine surface features into bulk metallic glass

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ABSTRACT

Molding of fine surface features into a bulk metallic glass (BMG) from the molten state was investigated. It was studied down to which a feature size in a mold could be reproduced in the BMG. A new casting apparatus was designed and built to carry out the experiments. The BMG used was Vitreloy 1™, $Zr_{41.24}Ti_{13.75}Cu_{12.5}Ni_{10}Be_{22.5}$, and it was observed to replicate mold features down to several nanometers. This has been verified with SEM photographs of both the mold surfaces and the BMG parts. Processing variables, including the injection temperature of the molten BMG and the atmosphere, in which it is molded, appear to have a great effect on the ability to reproduce the fine features on the mold surface.

INTRODUCTION

The random atomic arrangement of a bulk metallic glass (BMG) in the solid state lends itself to exploration of fine feature replication. The feature size created during a molding process should only be limited by the viscosity of the molten BMG and its surface energy relative to surface of replication. If the molten alloy were to crystallize during cooling, there would be an abrupt reduction in volume and features inherent to the crystal structure would emerge at the interface of the solidified alloy and the mold surface. The premise then, is that if the molten BMG could be made to flow into a mold feature, that feature should be reproducible almost to the atomic level.

EXPERIMANTAL DETAILS

A machine has been designed to address the two areas of concern; namely the viscosity of the molten BMG and the ability to overcome the surface energy at the flow front leading into the mold features to be replicated. These issues are addressed by moving the molten BMG from the heating zone and into the mold quickly, using a pneumatic injection cylinder, in order to minimize the temperature loss and resulting viscosity increase. After injection into the mold, a positive holding pressure is applied by the injection plunger used to force the melt into the mold in an effort to overcome the surface energy at the molten flow front (at the end of fill). Figure 1 shows the components of the machine. A copper mold is used because of its high thermal conductivity and acts as a heat sink for the rapid cooling of the BMG. The cavity dimensions are 3.18 x 12.70 x 19.05 mm (thick region) and 1.59 x 12.70 x 0.19.05 mm (thin region). The cavity size was influenced both by the coupling requirements of the induction heater used to melt the BMG and the size of the pre-fabricated BMG samples available for use in the test. The BMG used was Vitreloy1, $Zr_{41.24}Ti_{13.75}Cu_{12.5}Ni_{10}Be_{22.5}$ [1] provided by Liquid Metal Technology. Mold inserts (not shown) were created to fit into the mold surface. These inserts were polished and then scribed or indented using a diamond tip, which created the features to be replicated, as well as a grid for location purposes under the microscope. The mold is restrained inside of a stainless

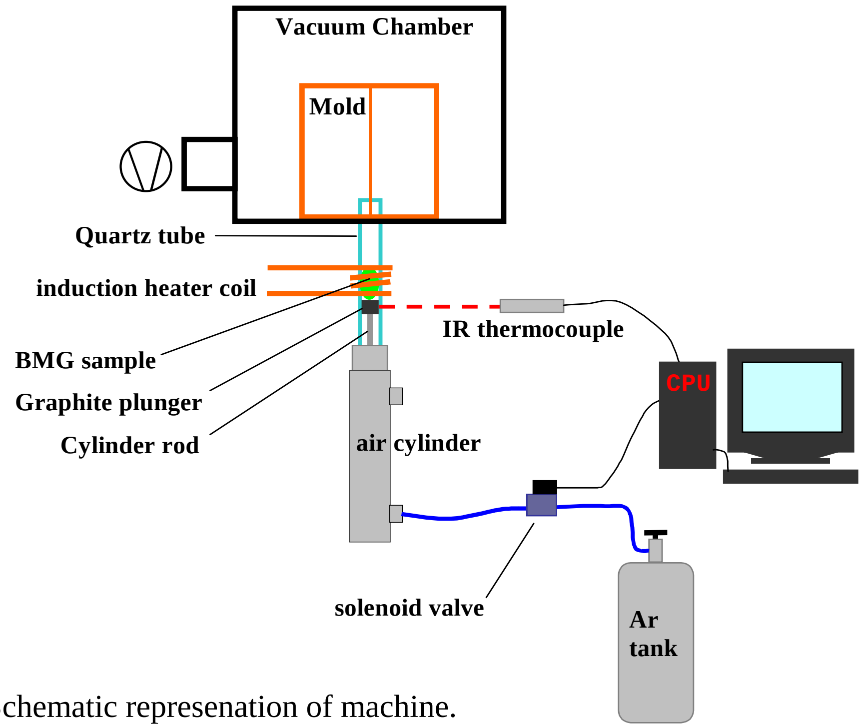

Figure 1. Schematic represenation of machine.

Figure 2. Photograph of apparatus with induction heater at back right.

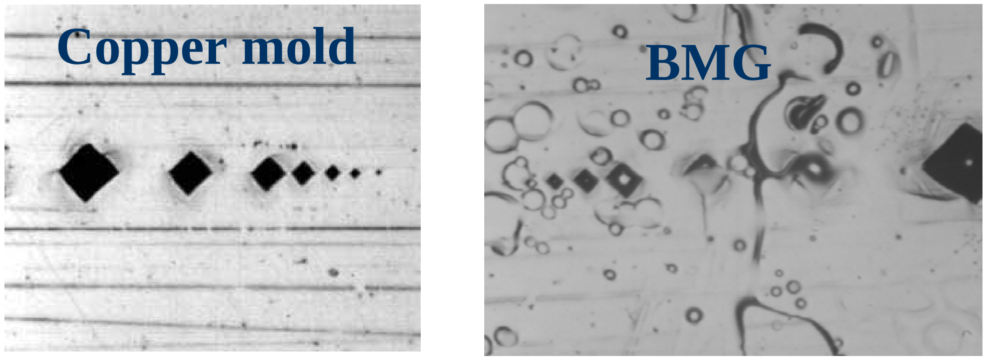

Figure 3. Micrograph of mold surface and features replicated in the BMG.

steel vacuum chamber, which has an entry port at the bottom where the injection barrel passes though and into the mold. The barrel is fused quartz tubing, and the injection plunger is machined graphite. A turbo molecular pump is used to evacuate the chamber and barrel and achieve a vacuum of 10^{-5} mbar. Figure 2 shows a photograph of the apparatus. The injection is controlled using computer software, which monitors the temperature of the molten BMG and triggers the injection once the desired temperature is reached. The induction heating method is ideal because it brings the BMG up to temperature quickly, which minimizes the reaction with the quartz barrel, reducing the potential introduction of contaminants that would act as nucleation sites upon cooling. The injection temperature is set at 1173 K, which is 150 K above the liquidus temperature of the BMG, to ensure dissolution of any heterogeneous nucleants that might be present above the melting point during heating.

DISCUSSION

Figure 3 shows a optical micrograph of the mold and the resulting replication in the BMG molding. It can be seen that some of the indentations have not replicated, while indentations on both sides of them have. A likely explanation for this result lies with the turbulent filling of the mold. The uneven flow over the surface would create a difference of velocities and affect the filling of the features. Figure 4 is a micrograph taken with a scanning electron microscope (SEM) of one of the replicated indentations. The shear bands, created in the copper by the indenter, have been replicated. These ridges of the bands are less than one micron in width. The tip of the pyramid is also less than one micron (not shown), but it can be seen that the tip has not filled. The melt cooled (viscosity increased) before reaching the point of the indentation. The shear bands indicate the ability of the BMG to replicate sub-micron features, but this replication is dependent upon the rate at which heat is removed from the melt. It would seem reasonable, then, that a higher mold temperature may allow the melt to cool more slowly, allowing the viscosity to remain relatively low and fill the pyramid completely.

Trials were run with the mold at several temperatures between 200°C and 350°C and melt temperature of either 900°C or 950°C. The best surface replication was seen at 275°C against both copper and stainless steel mold inserts. Figure 5 shows a series of photographs taken of the stainless steel insert and the resulting BMG molding. The diamond-scribed features appear rounded at the vertex because the focal plane is that of the flat mold surface. The width and depth of the scribed feature is 30 μm and 18 μm respectively. The tip of the feature has filled

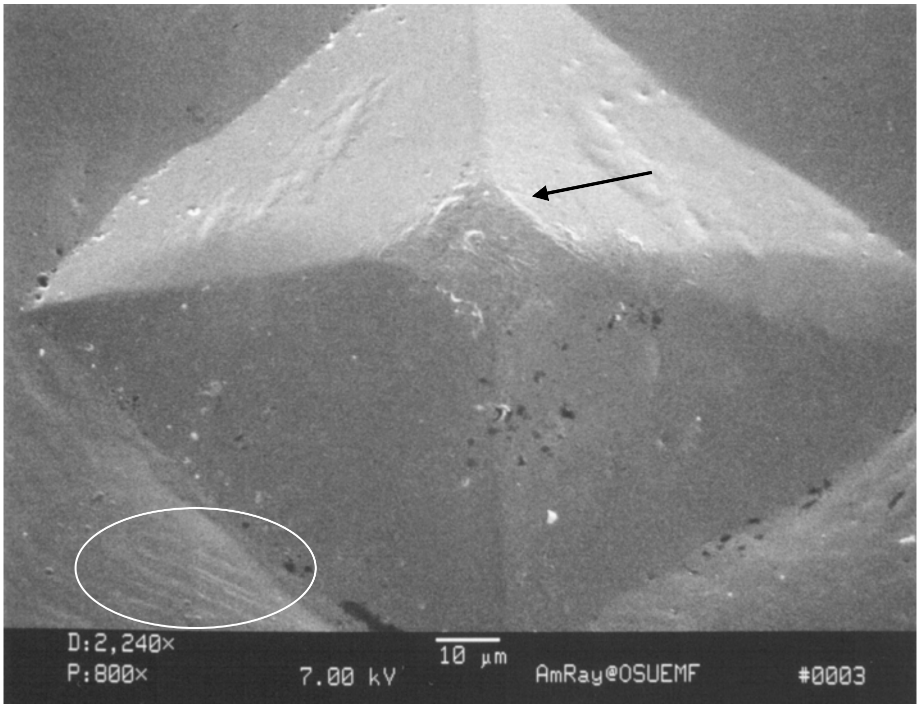

Figure 4. Picture of BMG. Shear bands were created in the copper by the diamond indenter. The circled area shows the bands, replicated in the BMG. The melt froze before entire fill of the indentation as indicated by the arrow.

completely. Furthermore, at the base of the feature, the surface of the insert was replicated, and sub-micron replication can be seen here. The two sets of arrows point to noticeable features on the insert and BMG cast part. Note that the magnification is slightly different between the two photos. To verify the BMG did in fact replicate the surface, a backscattered image of the casting was taken with the SEM. The lack of shade contrast on the surface of the BMG indicates that the material surface is in fact homogenous. The image is shown in Figure 6. The darker regions are merely shadows created by the tilt angle of the sample inside the SEM. From this, we conclude that the melt and mold temperature both play a role in determining the feature size that can be replicated because of the effect these temperatures have on the melt viscosity. Figure 7 is an SEM photograph of a BMG sample that has replicated machine marks on the copper mold surface. This picture represents the smallest replicated features observed in this study. The SEM magnification is 10,000x, and the feature size is of the order of 100 nanometers. There was a noticeable decrease in the ability of the BMG to replicate the mold surface when the chamber was backfilled with Argon gas, except in certain instances. The presence of the gas, however, effectively prevented the molten BMG from wetting to the quartz barrel during injection, thereby reducing failures by seizing. Two phenomena may be occurring here: first, the surface energies of the molten BMG and the contact are affected by the presence of the gas, thereby influencing the wetting of the two; second, the gas becomes trapped in the features and is unable to escape, which prevents complete fill. We are in the process of investigating this and determining the optimal processing environment, which is most likely a fractional pressure of inert gas within the chamber.

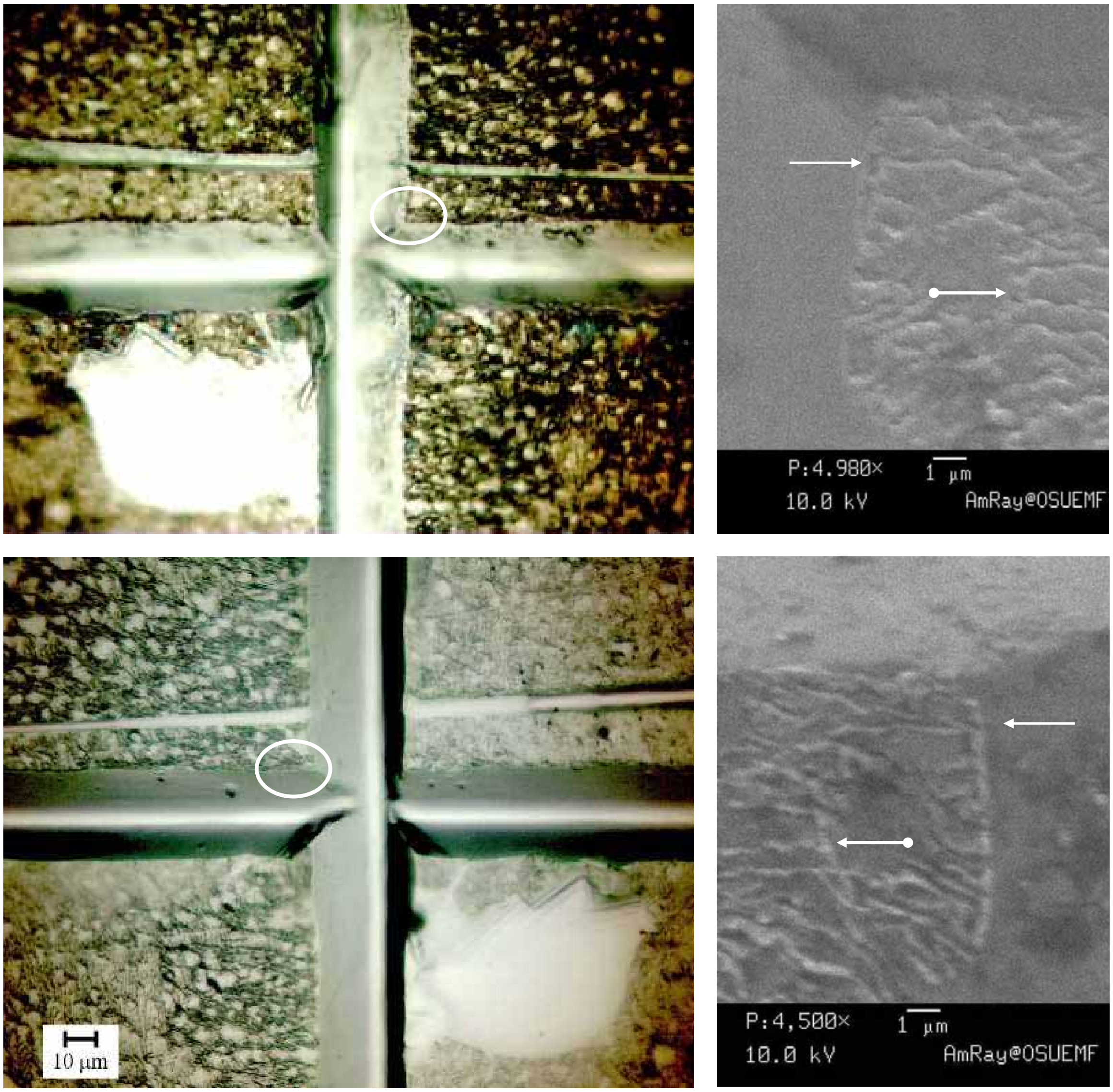

Figure 5. Photograph of mold surface (top two) and features replicated in the BMG (bottom two).

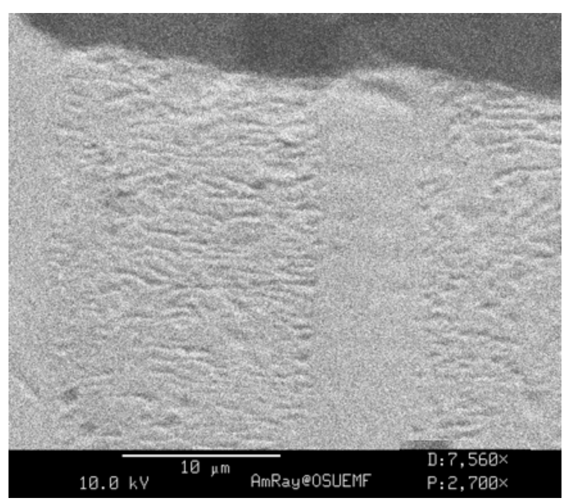

Figure 6. Backscattered image taken with a SEM to verify that the above casting surface was a homogenous replication of the steel insert. The darker areas are shadows.

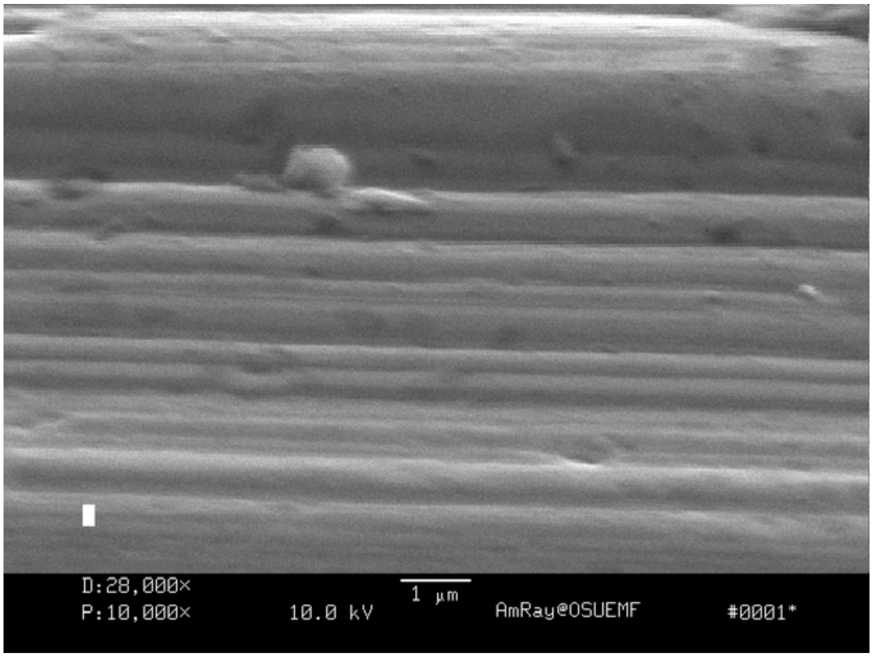

Figure 7. Replication of sub-micron features in BMG. Scale bar is 1 μm.

CONCLUSION

It has been shown that a BMG, formed from the molten state, is capable of reproducing surface features of a mold down to some 100 nanometers. The processing variables, which were seen to most significantly effect the replication, are mold temperature, melt temperature, and injection atmosphere. A more rigorous discussion of the machine, test results, and future work exists in the form of a thesis document [2]. Future work will concentrate on better defining the effect process variables, such as melt temperature, have on the size of features which can be replicated.

ACKNOWLEDGEMENTS

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