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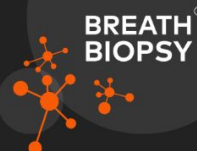
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## PAPER

## Controllable thermoplastic forming of bulk metallic glasses in milliseconds by resistance welding forming

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**Abstract**

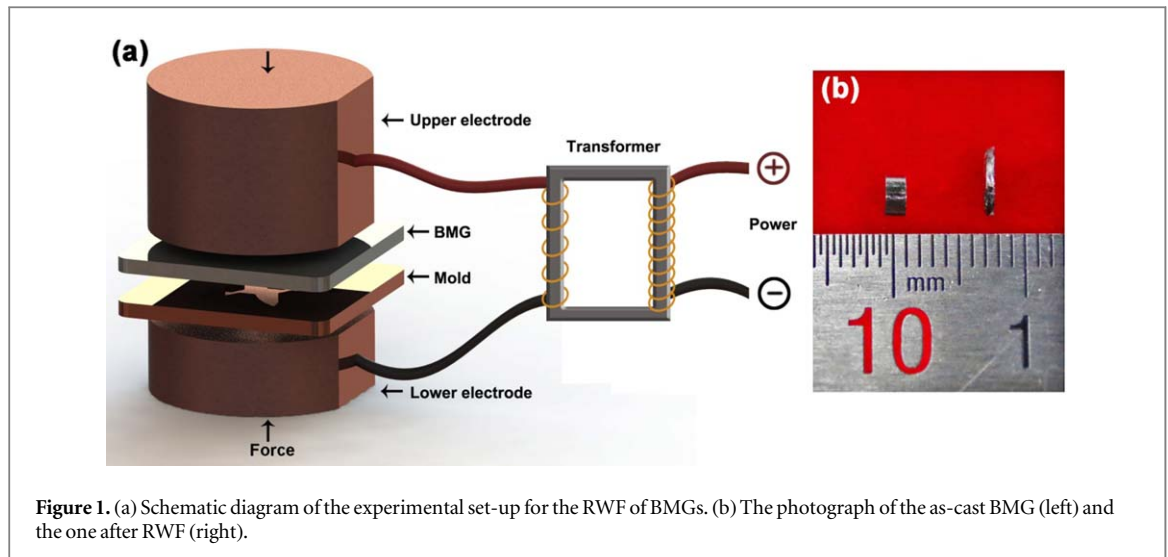
The work proposed a controllable and rapid thermoplastic forming approach—the resistance welding forming (RWF) method for bulk metallic glasses (BMGs). The rapid forming approach can finish the thermoplastic forming of BMGs in 150 milliseconds without crystallization and degradation in mechanical performance. Besides, the RWF is also proved to be competent in the fabrication of parts and structures with the length scale ranging from macro scale to micro scale. Our results may have promising applications in the rapid fabrication of macro to micro scale products and devices using metallic materials.

**1. Introduction**

The of bulk metallic glasses (BMGs) have triggered a lot of significant interests since their discovery [1–9] owing to their unique properties such as high strength, high specific strength, large elastic strain limit, and excellent wear and corrosion resistances *et al.* Specifically, by virtue of being glasses, they get soft when heated into the supercooled liquid region (SLR), hence, viscoplastic shaping can be carried out [10–12]. It should be noticed that due to inherent homogeneity of the amorphous structure, they exhibit high dimensional accuracy during the thermoplastic forming [13–16]. Therefore, BMGs have promising applications in the fields of Micro-electromechanical Systems (MEMS), micro mold inserts, biomedical devices and implants, micro robotics, micromanipulators and so on [17, 18]. However, this potential application is practically limited by the rapidly intervening crystallization of the relaxed ‘supercooled’ liquid at elevated temperature. In the past thermoplastic forming methods, the thermoplastic forming of BMGs usually occurs in three separate steps of heating, force applying and quenching [10]. During these separated steps, much prime time was wasted and the BMGs would be in huge risk of being crystallized or oxidized. Therefore, to develop novel thermoplastic forming method to bypass crystallization has become a key issue for the applications of BMGs.

An appropriate heating method providing a fast homogeneous and volumetric heating is Joule heating [19]. Johnson *et al* [20] have achieved the thermoplastic forming of BMGs at a heating rate of about  $10^6$  Ks<sup>-1</sup> by using a rapid capacitor discharge. By coupling such a capacitor discharge with an applied static magnetic field, BMGs can be brought to a low-viscosity state while exploiting the accompanying Laplace force to form the sample to a net shape, all while avoiding crystallization [21]. Based on this technique, quantitative descriptions of the rapid capacitive discharge process and a thorough analysis of the physical properties of the metallic glass over the inaccessible temperature regime are investigated [22]. However, the parameters (e. g. voltage, electric current) in the capacitive discharge process are difficult to be precisely controlled, which makes the capacitor-discharge heating quite complex [23]. Within the ultra-short discharge time window, the electric field and current are varying rapidly, which brings about electromagnetic effects that can cause non-uniformities in the sample, such as eddy currents, which are induced when a conductor is exposed to a changing magnetic field. Therefore, it is difficult to guarantee the forming of BMG is uniform and the product has a good quality by this method [23].

In present work, we propose an advanced Joule heating technique—the millisecond resistance welding forming (RWF), and apply it to the forming of BMGs, thereby producing net-shaped metallic glass macroscopic



parts and micro patterns with high quality. In contrast with the capacitive discharge forming, the electric current stands at a constant value during the RWF, making it a convenient and controllable method for the millisecond thermoplastic forming of BMGs.

Resistance welding is a welding technology which is widely used in manufacturing industry for joining metal sheets or components. The welding is performed by conducting a strong current through the metal combination to heat up and finally melt the metals at localized point(s) of the workpieces to be welded [24–26].

## 2. Experimental

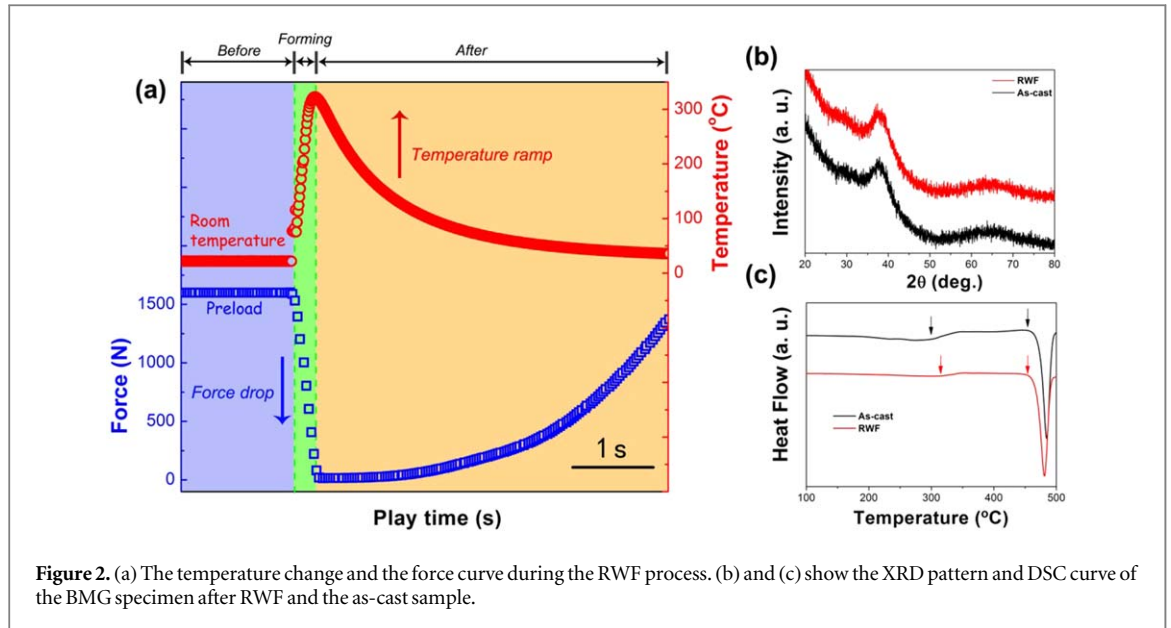
The  $Zr_{35}Ti_{30}Cu_{8.25}Be_{26.75}$  BMG cylinder samples were chosen for present research. For the convenience of RWF, the cylinder sample with a diameter of 5 mm was cut into a thickness of 1.4 mm and then the surface was polished by the abrasive paper and polishing machine. The amorphous nature of the Zr-based metallic glasses were ascertained by x-ray diffraction (XRD, Bruker D8 Advance) with  $Cu K_{\alpha}$  radiation. The glass transition temperature  $T_g$  and crystallization temperature  $T_x$  of the as-cast sample are 305 °C and 453 °C, which is confirmed by the differential scanning calorimetry (DSC; Perkin–Elmer DSC-8000) at a heating rate of 20 K  $min^{-1}$ . The specific heat is also measured by the DSC using sapphire as the standard material. The micro morphologies were collected on a scanning electron microscope (SEM; Hitachi SU-70) instrument. The nano-scale mechanical performance was tested on a nanoindentation testing system (Hysitron TI950).

The experimental set-up for the RWF of BMGs is schematically shown in figure 1(a). The Zr-based BMG and the mold were firstly stacked between the upper electrode and the lower electrode, which was made by copper to enhance the electric conductivity. After that, a preload force of 1600 N was exerted upon the electrodes to make the BMG and the mold have a tight contact and provide the required force for the RWF. Finally, the electric current of 1500 A was applied for 150 milliseconds through the transformer, the temperature of BMG increased into its SLR and got soft in a flash because of the Joule heating effect. The viscous BMG filled into the mold and formed specific structures under the action of force. As we know, the thermoplastic forming of BMGs always involves heating. As a metastable material, BMGs tends to transform from amorphous to crystalline state under elevated temperature, and the efficient strategy would be shortening the processing time as possible as one can. However, in present RWF process, the heating and forming work simultaneously in an instant, saving much prime time to avoid crystallization of BMG.

## 3. Results and discussions

Figure 1(b) presents the photograph of the as-cast BMG (left) and the one after RWF (right), one can see that the thickness of the sample has an obvious change from 1.4 mm to 0.65 mm, indicating the BMG gets soft during the RWF.

The temperature change and the force curve during the RWF process are presented in figure 2(a). Before the RWF started, the temperature stayed at room temperature and the force held at the preload force. When the RWF begun, the temperature ramped into the SLR of BMG in a very short time at a heat rate of about 1103 °C  $s^{-1}$  the peak value reached 322.8 °C. At the same time, the force dropped sharply, indicating the BMG



**Figure 2.** (a) The temperature change and the force curve during the RWF process. (b) and (c) show the XRD pattern and DSC curve of the BMG specimen after RWF and the as-cast sample.

got soft with the increase of temperature. During this period of time, the viscous BMG filled into the mold cavity and the structure was formed on its surface. It can be seen from figure 2(a) that the temperature ramping process lasts for about 270 ms, nearly the same time span with the force dropping process, which means the two procedures are linked with each other. When RWF begins, temperature is a fast gradient process from the electrode to the interface between the electrode and BMG and then to the forming surface. Therefore, when RWF begins, the contact surface between the electrode and BMG enters a viscous state in a very short time, which results in the decrease of the preloading force almost at the same time. Then the viscous state goes through the same process during the transfer of temperature from the contact surface to the forming surface, which leads to the continuous decrease of the preloading force in the forming process. Therefore, it can be seen that the two procedures are linked with each other.

To further study the intrinsic nature of the formed BMG, figures 2(b) and (c) show the XRD pattern and DSC curve of the BMG specimen after RWF and the as-cast sample. Both the RWF-treated and as-cast BMG samples show typical amorphous amorphous peak in the XRD pattern. The results prove that the formed BMG remains its amorphous nature. That is to say, by uniformly heating a bulk metallic glass in milliseconds, the stability of the supercooled liquid against crystallization is dramatically extended. The glass transition temperature  $T_g$  and crystallization temperature  $T_x$  of the as-cast sample are 305 °C and 453 °C, in comparison, the corresponding values of the RWF treated specimen are 310 °C and 453 °C, which can be seen from figure 2(c). Only small differences of thermal parameters between the RWF-treated and the as-cast BMG samples can be found.

The principle of resistance welding is the Joule heating law where the heat  $Q$  is generated depending on the Ohm's law as expressed in the following formula:

$$Q = I * U * \tau = I^2 * R * \tau \quad (1)$$

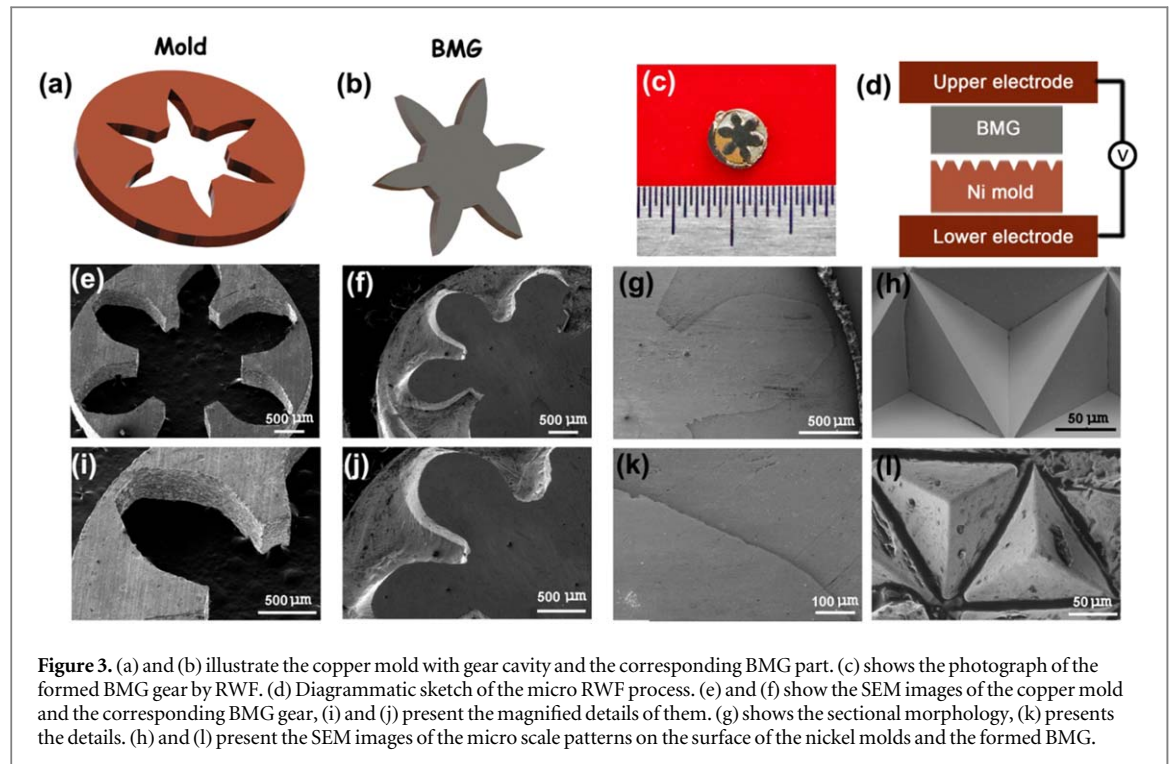
In equation (1),  $I$  and  $U$  are the current and voltage passing through the specimen,  $R$  is the resistance of the base substrate, and  $\tau$  is the duration time of the current flow. In the present case,  $I = 1500$  A,  $\tau = 0.15$  s. The electrical resistivity  $\rho$  of metallic glasses is  $\sim 100$  to  $250 \mu\Omega \cdot \text{cm}$  [27], therefore, the resistance of the BMG sample:

$$R \geq \rho * L/S = \frac{100 \mu\Omega \cdot \text{cm} * 0.14 \text{ cm}}{\pi * (0.25 * 0.25) \text{ cm}^2} = 71.34 \mu\Omega \quad (2)$$

That is to say, the energy provided by the Joule heat effect  $Q \geq 23.96$  J according to equation (1). On the other hand, according to the heat equation:

$$E = m * C * \Delta T \quad (3)$$

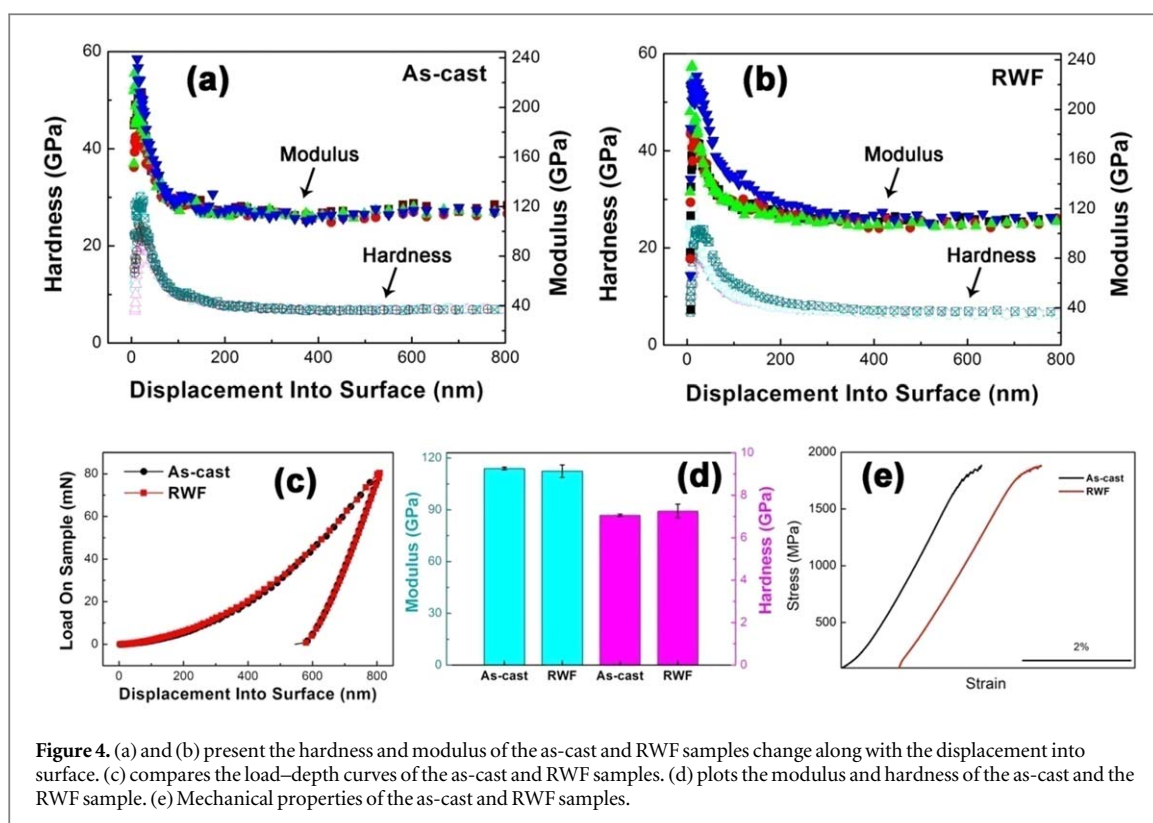
where  $m$ ,  $C$  and  $\Delta T$  are the mass, specific heat capacity and temperature raise of an object when given a heat  $E$ . Here, the values of  $m$  and  $C$  were measured to be 0.15 g and 0.417 J/(g · K). To ensure the processed Zr-based BMG reaches the SLR state, the temperature rise  $\Delta T$  should be 280 °C at least (room temperature = 25 °C). Therefore, the heat quantity  $E$  needed to drive the BMG sample into its SLR would be about 17.51 J according to the equation (3). Here, we can see the Joule heat  $Q$  is greater than the required heat quantity  $E$ , indicating the Joule heat could provide enough energy for the BMGs to get soft.



The BMGs were considered as the ideal candidates for the fabrication of cross-scale products and devices [10]. In present work, structures from macro scale to micro scale were tried to fabricate on the surface of BMGs by using the proposed RWF method. A preload force of 1600 N and a electric current of 1500 A were applied to the BMG sample through the electrodes, causing the BMG sample to get soft and fill into the mold, and the whole forming process lasts for only 270 ms. During the macro scale fabrication, the molds were made of copper plate which was cut into the gear shape by the wire electrical discharge machining (EDM). The gear has six teeth and the diameter of its addendum circle is 4 mm, as illustrated in figure 3(a). The formed BMG product is also illustrated in figure 3(b). Figure 3(c) shows the photograph of the formed BMG gear by RWF. In the case of microscale fabrication, the molds were substituted by the nickel mold with periodic pyramid patterns on it. The side length of the regular triangle is  $170\ \mu\text{m}$ , and the diagrammatic sketch of the micro RWF process is drawn in figure 3(d).

Figures 3(e) and (f) show the SEM images of the copper mold and the corresponding BMG gear, respectively, and figures 3(i) and (j) present the magnified details of (e) and (f). One can see that the BMG fully replicated the gear structure in the copper mold cavity. Figure 3(g) shows the sectional morphology, and the detail is presented in figure 3(k). It can be clearly seen that the BMG completely filled into the mold cavity, the boundary line of the BMG product fits well with that of the copper mold. In addition to the macro forming, the RWF can also be used to fabricate micro patterns on the surface of BMG. Figures 3(h) and (l) present the SEM images of the micro scale patterns on the surface of the nickel molds and the formed BMG patterns on it. The side length of the pyramid cavity on the nickel mold is  $170\ \mu\text{m}$  and the side length of the pyramid bulge on the BMG replica is  $165\ \mu\text{m}$ , with only a small dimension mismatch of about 3%, indicating that the RWF method is feasible to fabricate the controllable micro structures on the surface of BMGs. These results indicate that the fabrication of macro-scale structures or parts as well as micro patterns on BMGs could be achieved by the effective RWF.

In order to investigate if there is any change in local mechanical performance during the RWF process, the as-cast and RWF samples were tested by nanoindentation. Figures 4(a) and (b) present the hardness and modulus of the as-cast and RWF samples change along with the displacement into surface. Through repeated measurement, the data curves show good uniformity. The load–depth curves of the as-cast and RWF samples are compared in figure 4(c). Obviously, they reveal almost the same mechanical behavior, indicating the RWF did not bring in the change of mechanical performance. The measured hardness ( $H$ ) and elastic modulus ( $E$ ) of the as-cast sample are  $H = 7.06 \pm 0.05\ \text{GPa}$  and  $E = 113.9 \pm 0.8\ \text{GPa}$ , while for the BMG sample,  $H = 7.26 \pm 0.32\ \text{GPa}$  and  $E = 112.4 \pm 3.6\ \text{GPa}$ . The histogram in figure 4(d) plots the modulus and hardness of the as-cast and the RWF BMG sample. The mechanical properties of the as-cast and RWF samples were also measured, the results are presented in figure 4(e). It is obvious that the two samples show the similar mechanical performance, which indicate that the RWF method would not bring the decay of the mechanical properties.



**Figure 4.** (a) and (b) present the hardness and modulus of the as-cast and RWF samples change along with the displacement into surface. (c) compares the load–depth curves of the as-cast and RWF samples. (d) plots the modulus and hardness of the as-cast and the RWF sample. (e) Mechanical properties of the as-cast and RWF samples.

## 4. Conclusions

To sum up, the developed RWF method in present research provides a controllable millisecond thermoplastic forming approach for BMGs. The rapid forming method (less than 150 ms) saves prime time during the thermoplastic forming of BMGs to the maximum extent, avoiding crystallization and oxidization. Based on this method, macro gear parts and micro pyramid patterns were successfully fabricated. The intrinsic structure of the RWF sample was found to keep amorphous and the mechanical performance did not degrade after the forming process. Our results provide a new route for the rapid fabrication of macro to micro products and structures on the surface of BMGs and could greatly extend the promising applications of this materials.

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