



# Superplastic diffusion bonding of metallic glasses by rapid heating

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## ABSTRACT

We show a superplastic diffusion bonding method to join metallic glasses (MGs) via high-speed heating at 2000 K/min taking a timescale of the order of microseconds to milliseconds at ambient pressure because the minimum viscosity of supercooled liquid drastically decreases as the heating rate increases. The results show strong metallic bonds are formed but the amorphous structure is still maintained. Finally, we establish a general equation containing the involved parameters for high-speed heating bonding. This method provides a new path to join MGs rapidly and benefits for improving the critical size of MGs.

## 1. Introduction

Metallic glasses (MGs) have widely potential application in fields of sports, machinery, medical, electronics, chemical industry, aviation, aerospace and others because of their excellent properties of mechanics, physics, chemistry and biology [1,2]. However, the poor glass forming ability limits commercial applications of MGs. Therefore, joining MGs is presently attracting immense research interest on the global level. For any kind of material, how to join them depends on their own physical properties. Due to the antioxidant ability and relatively wide supercooled liquid region, polymers can be easily spliced by thermoplastic deforming or melting of the interface [3–5]. Easy oxidation and high operation temperature limit the thermal bonding in conventional alloys. Interestingly, MGs have also extraordinary supercooled liquid region like plastics [6]. This phenomenon has been reported in many kinds of MGs, such as  $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$  [7],  $Pd_{40}Cu_{40}P_{20}$  [8] and  $La_{55}Al_{25}Ni_{20}$  [9]. The use of viscous flow of the supercooled liquid for forming processes is called superplastic forming, and has been widely used in the manufacturing [10]. The components of MGs have directly been shaped by the superplastic forming [1,11,12]. A novel manufacturing technology, superplastic diffusion bonding (SP/DB), was developed when superplastic was combined with atom diffusion, giving rise to both cost reduction and weight saving compared to conventional manufacturing methods. More and more researchers preferentially use this method to join MGs.

However, a common problem for SP/DB methods is the high bonding viscosity of  $\sim 10^6 Pa s$  [13], which results in “violent” bonding pressure ( $\sim 10^2 MPa$  magnitude) and long bonding time ( $\sim 1 h$  level) [14–18]. Besides, avoiding crystallization has been mainly challenging since the diffusion kinetics and crystallization kinetics are similar. As a

result, this technology cannot be applied to some “mild” manufacturing environment, such as 3D printing, micro welding and so on. Obviously, neither high bonding pressure nor long bonding time is allowed in these manufacturing environments. To explore a new bonding way under the conditions of low pressure and short time, it is necessary to further study the physical properties of the MGs, look for new ways to lower processing viscosity and determine the bonding strategy.

In this paper, according to dramatical drop of minimum viscosity in supercooled liquid region and broadening of supercooled liquid region as heating rate increases [19], we propose a novel SP/DB method to join MGs through high-speed heating with bonding viscosity of  $10^3 Pa s$  magnitude. The operation time is from microseconds to milliseconds at ambient pressure. In our experiment, bonding tests were carried out under the condition of high heating rate at 2000 K/min using elaborately self-made experimental apparatus. Subsequently, the resulting bonds and bonding interfaces were mechanically tested by nanoindentation instrument and investigated by scanning electron microscopy (SEM) respectively. The results show strong metallic bonds are formed but the amorphous structure is still maintained. Finally, we established the general equation consisting of the involved parameters for seamless bonding. The SP/DB method provides a new path to improve the critical size of MGs and also benefits for fabricating MGs products with complex shape.

## 2. Experimental procedure

The material used in the present study is composed of  $41.2Zr-13.8Ti-12.5 Cu-10Ni-22.5Be$  (vit1) bulk metallic glass having a diameter of about 5 mm prepared by copper mold suction casting method. Structural analysis was performed by means of differential scanning

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calorimeter (DSC) and X-ray diffraction (XRD) in BRUKER D8 Advance using CuK $\alpha$  radiation.

SP/DB test specimens were fabricated from the as-cast metallic glass by means of electrical discharge machining. These specimens have a gauge diameter of 5 mm and thickness of 1 mm. The surface of amorphous structures is very stable and non-reactive. Therefore, in order to create a pristine surface, the test specimens were polished by 2500 (10 $\mu$ m) SiC sanding paper before the SP/DB tests.

A pair of specimens were overlapped and installed in elaborately self-made experimental apparatus which has a high heating rate of about 2000 K/min and heating temperature range from 508 K to 1273 K. The apparatus has been shown to be effective for heating specimens fast and avoiding temperature overshoot and oxidation of specimens. The details of the heating process are as follow. Metal liquid is fabricated as constant heat reservoir, and then overlapped specimens packed by a clamping device are put into the molten metal liquid. Specimens absorb heat through wall of the clamping device and reach thermal equilibrium with the liquid metal in a very short period of time. SP/DB tests were carried out in bonding pressures of 0.1 MPa for thermoplastic compression between the two specimens. Bonding temperature and heating-plus-holding time are shown in Table 1.

Cross-section of the interface in bonded specimens was polished and investigated by SEM. The bonding quality under different bonding conditions was further assessed by nanoindentation test to determine the hardness in the vicinity of the interface. 9 in-line indents spaced 20 $\mu$ m apart were performed across the bonded interface. The same indents were also performed along the bonded interface. Moreover, structure of the bonded specimens was confirmed by XRD observation.

### 3. Results

The purple curve in Fig. 1(a) is X-ray diffraction pattern of as-cast specimen, which indicates only a broad diffused peak without any detectable crystallized peaks. Fig. 1(b) shows DSC scans at 20 K/min of the as-cast specimen. As is known from the diagram, the glass transition temperature  $T_g$ , crystallization temperature  $T_x$ , width of supercooled liquid region  $\Delta T$ , solidus temperature  $T_s$  and the liquidus temperature  $T_l$  of vit1 are respectively 618 K, 688 K, 70 K, 925 K and 1005 K.

MGs will transfer to more stable crystalline phase in the heating process. So it is necessary that the structure of the specimens be confirmed by XRD observation after SP/DB tests. The black, red and blue curves in Fig. 1(a) are respectively X-ray diffraction patterns of bonded specimen 1, bonded specimen 2 and bonded specimen 3. These diffraction spectrums all show diffuse “steamed bread peak”. It can be concluded that they all present well amorphous structure.

Fig. 2 shows SEM of the polished cross-sectional area of the interface under different bonding conditions. Discontinuously filamentary interfaces can be observed, indicating the achievement of good joining between two specimens. Nanoindentation test results performed across and along the bonded interface are respectively shown in Fig. 3(a) and (b). They both reveal negligible hardness variation within experimental error. W. Chen et al. think a strong metallurgical bond formed, that is, comparable with the bulk,  $\tau_{\text{bond}} \approx \tau_{\text{bulk}}$  in the bonded region where the nanohardness values show negligible hardness variation. Based on the assumption, they established a simple model predicting the experimental relationship between the joint shear strength and the joining strain reasonably well [13]. Therefore, nanohardness test is effective for

proving joint strength of the bond. Compared with W. Chen, we had tested the nanohardness values both across and along bonding interfaces, as shown in Fig. 3. The nanohardness values show negligible hardness variation, further confirming a strong metallurgical bond in the bonded region comparable with the bulk.

### 4. Discussion

The mechanism of the introduced SP/DB method is theoretically described as follow. The supercooled liquid deforms or flows under an exerted stress. The existing cavities between interfaces will be filled with flowing pristine material under the exerted pressure. When pristine material from both sides of the interface approaches atomic contact a metallic bond forms. The timescale for forming metallic bond in this method is decided by the time it takes for the flowing pristine material to fill the cavity between interfaces. The filling time  $t$  is function of the oxide film thickness  $N$ , the viscosity of supercooled liquid  $\eta$ , the wetting of supercooled liquid on the oxide, the average length scale of the re-filled material  $d$ , the forming pressure  $\sigma$  and temperature  $T$  for typical processing conditions [13]. The time to establish a metallic bond can be estimated by a modified Hagen–Poiseuille relation: [20].

$$t = \frac{32N^2\eta}{\sigma d^2} + \frac{\alpha N\eta}{\sigma d} \quad (1)$$

The flow stress  $\sigma$  is estimated with

$$\sigma = 3\eta\dot{\epsilon} \quad (2)$$

under the assumption of Newtonian flow. From equations (1) and (2), we can obviously find that bonding time and pressure are both controlled by the viscosity of the supercooled liquid. The smaller the viscosity is, the shorter the bonding time becomes and the smaller the bonding pressure will be. In supercooled liquid region, viscosity declines rapidly as the temperature increases and their relationship can be described by the VFT equation,

$$\eta = \eta_0 \exp\left(\frac{D^*T_0}{T - T_0}\right) \quad (3)$$

in which  $\eta_0$  is the high temperature limit of the viscosity,  $T_0$  is VFT temperature, and  $D^*$  is fragility parameters.  $\eta_0$  can be described by the following relation,

$$\eta_0 = \frac{N_A h}{V} \quad (4)$$

in which  $N_A$  is the Avogadro constant,  $h$  is the Planck constant and  $V$  is the free volume.

However, unlike plastics, supercooled liquid region of MGs is in fact astatic. This is a very interesting characteristic underutilized by many researchers. Supercooled liquid region moves to high temperature region and meanwhile its width also broadens as heating rate increases [19]. According to equation (3), increasing the heating rate theoretically makes minimum viscosity in supercooled liquid region decline because of its variation trend. Minimum viscosity can be described as

$$\eta_{\text{min}} = \eta_0 \exp\left(\frac{D^*T_0}{T_x - T_0}\right) \quad (5)$$

in which  $\frac{dT}{dt}$  determines  $T_x$ . This means that we have a wider viscous range to join MGs. Increasing the heating rate, therefore, is an effective way to realize rapid SP/DB in supercooled liquid region. The rapid SP/DB by the use of viscous flow of the supercooled liquid must be implemented under the condition where the low viscosity for easy deformation and the short time available for crystallization have to be simultaneously satisfied.

Table 2 lists glass transition temperature, crystallization temperature of vit1 and its width of the supercooled liquid region corresponding to different heating rates [19]. The minimum viscosity of vit1 corresponding to different heating rates can be estimated according to

**Table 1**

The experimental parameters combination.

No.	Temperature(K)	Time(s)
1	770	90
2	773	45
3	783	30

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