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Characterization of shear transformation zone volume during Orthogonal micro turning of Zr-based Bulk metallic glass

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Abstract

Bulk metallic glasses (BMGs) have unique superior properties as compared to their crystalline counterparts because of the disordered structure at the atomic scale. This study presents the experimental work on Zr-based BMGs during orthogonal micro-turning. The shear transformation zone (STZ) volume of the strained surface generated during machining was characterized by nanoindentaion technique. The STZ volume was found to be lower for the machined region than the unmachined region for all the cases of cutting speeds and feed values which suggest the densification of the atoms at the machined surface due to combined influence of machining and nanoindentation. The densification of atoms suggests the work hardening (strain hardening) phenomenon which is generally not visible for amorphous metallic alloys.

Keywords: Shear transformation zone (STZ), Bulk metallic glass, Micro turning, Nanoindentaion

1. Introduction

Machining of Bulk metallic glasses (BMGs) retains some distinctive challenges because of their high strength, high elastic limit (~2%), high hardness and low thermal conductivity, excellent corrosion, and wear resistance. Amorphous nature of BMGs can be attributed to the random arrangements of atoms, which contrasts with the crystalline materials that possess systematic long-range arrangements of atoms. Since, the BMGs are relatively new class of material as compared to other crystalline alloys (Ti-alloys or Ni-alloys), hence their response under different manufacturing and fabrication techniques are still under investigation. Machining is one of the most important process to fabricate high precision components with high dimensional accuracy.

The hardness is an important parameter that needs a detailed investigation to understand the influence of machining parameters on the machined surface. The extent of hardening or softening in BMGs depends upon the amount of free volume produced during deformation. The plastic deformation of metallic glasses occurs through the formation of shear bands. The structural state of an amorphous alloy is dependent on the free volume which is an intrinsic parameter. When an external shear stress is applied to the material, there would be a change in the free volume of the material as the atoms rearrange themselves collectively to form small group of atoms or zone called as the shear transformation zones (STZ). It can be further confined as STZs are basically collective rearrangements of atoms formed by local hopping of atoms to form a small cluster of atoms under the influence of an external shear stress [1]. The concept of STZ was fundamentally proposed by Argon [2] and they are known as the basic unit of plasticity carriers during the deformation in metallic glasses. For an STZ event to occur in metallic glasses, free volume is the nucleation site. STZs will be triggered in the region where the free volume is more, and its operation depends completely on the amount of free volume present in the region. Consequently, this local rearrangement of atoms involves an elastic reconfiguration of atomic neighbours and thus redistribution of free volume which can generate hardening or softening in a metallic glass under the response of an applied shear stress. The STZ size and volume plays an important role to understand the plasticity of metallic glasses [1-2]. However, it is difficult to experimentally quantify the STZ volume due to its operation at the atomic level. The atomic rearrangement is extremely difficult to capture experimentally especially after aggressive machining conditions. The simulation studies on metallic glasses for the STZ event throws more light on the plasticity of metallic glasses but fails to capture the hardening or softening behaviour of metallic glasses. A rigorous experimental characterization of STZ event after micromachining has not been reported in the literature. Nanoindentation is an attractive technique for these kinds of studies which uses extremely small volume of material to study the localized plastic deformation [3] and it can capture the changes on the machined sub-surface, which is otherwise not possible with the micro-indentation technique.

The objective of this study is to investigate the effect of micro turning parameters on the STZ volume of the machined region, which was further compared with the STZ volume of the unmachined region. The characterization of the STZ volume was done by constant strain rate method using nanoindentation.

2. Experimental Details

2.1. Machining Experiments

Orthogonal micro turning operation were conducted on circular disk of Zr-BMG ($Zr_{67}Cu_{10.6}Ni_{9.8}Ti_{8.8}Be_{3.8}$ (wt%)). The commercial name of the Zr-BMG used was Vit 1b-X. The diameter of the work piece was 9mm and the thickness was 0.8mm. The edge radius of the cutting insert was ~15µm. The experiments were conducted on the high precision CNC machining center (MikroTools DT 110i). The work piece was held in the spindle with the help of a cylindrical fixture. The rotational motion of the fixture attached with the spindle provides the necessary cutting speed required for grooving operation. The tungsten carbide cutting inserts with PVD coating (TiAlN) was used for the experiments. The rake angle and the clearance angle of the inserts were 0° and 10° respectively. Cutting insert attached with the tool holder was mounted on the dynamometer (Kistler MiniDyne 9256 C1).

Table 1: Machining Parameters

Cutting speed (m/min)	84, 56, 28
Feed (µm/rev)	10, 20, 30, 40

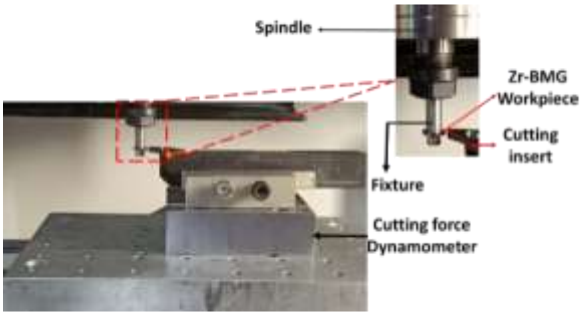

Fig. 1. Experimental set up for micro turning.

The machining parameters used in the experiments are mentioned in the Table 1. Each parameter was varied one at a time, and hence a total of 12 experiments were conducted. The cutting and the thrust forces generated during the machining process were captured using a dynamometer. The cutting operation was done at room temperature without any use of the coolant. Each experiments were repeated 3 times and average values were used for the analysis.

2.2. Nanoindentaion Experiments

The nanoindentation measurements were done on TI Premier (Bruker). The tests were done with a Berkovich indenter of tip radius approx. 150 nm and the half angle of 65.3°. The area function of the tip was calibrated with a known standard fused quartz. Oliver Pharr method was used to calculate the hardness (H) and reduced modulus (E_r) from the load-displacement curve by [3],

$$H = \frac{P_{max}}{A_C} \quad (1)$$

$$E_r = \frac{1}{\beta}\frac{S\sqrt{\pi}}{2\sqrt{A_C}} \quad (2)$$

where P_{max} represents the maximum force expressed in micro newtons or milli newton and A_c is the area of the contact. Stiffness (S) of the material can be determined from the slope of the load-displacement curve from the initial elastic part of the unloading curve. β acts as geometrical constant and is equivalent to 1.

The area function of the Berkovich indenter is estimated using the equation,

$$A\,(h_c) = C_0 h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + C_3 h_c^{1/4} + \ldots\ldots\ C_8 h_c^{1/8} \quad (3)$$

where $A\,(h_c)$ is the area function which is equated from C_0=24.5 (Berkovich indenter) and C_1 to C_8 are constants and hc is the contact depth measured during Nanoindentation. The drift rate was maintained below 0.05 nm/s for all the tests. The tip was calibrated regularly to avoid any tip rounding effect. The samples were polished with 2000 grade polishing paper and then to obtain mirror finish diamond polishing was done. The samples were sonicated for 10-15 mins after polishing to clean the surface.

All the tests were done in constant strain rate ($\dot{P}/P$) method. In this method, ($\dot{P}/P$) is maintained constant where $\dot{P}$ = (dP/dt) by penetration of the indenter in the specimen at constant strain rate (where $\dot{P}$ or $^{dP}/_{dt}$ is the indentation loading rate and P is the indentation load). The maximum load used for the strain rate test was 10000 µN. The load increases exponentially with time. The tests are done on five different strain rates, viz. 0.025 s^{-1}, 0.075 s^{-1}, 0.25 s^{-1}, 0.75 s^{-1} and 1 s^{-1}. A series of ten indentations at each strain rate were done on the machined surface and the unmachined region. On the machined surface, the indentations were made within the range of 1 µm to 4 µm distance from the machined surface at each strain rate. On the unmachined region, the indentations were done at each strain rate and the spacing between the indents was kept as 30 µm (Fig. 2).

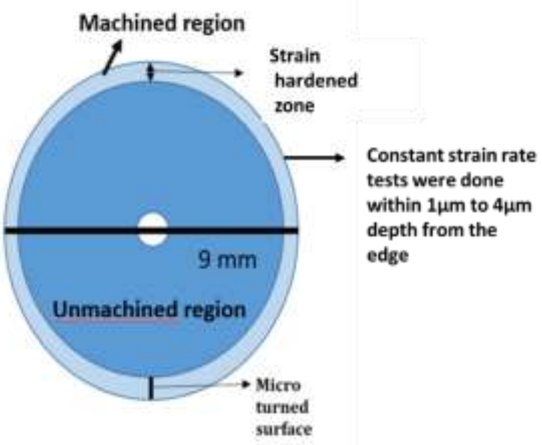

Fig.2 Schematic representation of the Nanoindentation zone

3. Result and discussion

Shear transformation zones are basically clusters of atoms formed under the influence of an applied stress. The plastic deformation in metallic glasses occurs through the cooperative shearing leading to the formation of STZs which further nucleates into shear bands. STZs are immobile; their formation will be more prone in the region where the free volume is in excess. The surrounding matrix in a metallic glass is changed even if a single STZ is created. Due to the creation of the first STZ, new free volume would be generated and thus it would attract more STZs events in the presence of local strain field. When the free volume is in excess, then larger number of STZ events would be created. The free volume annihilates through diffusional relaxation process, the atoms try to rearrange themselves in ordered manner [1-2]. The two main factors which are primarily responsible for the deformation in metallic glasses is firstly the free volume accumulation which results in softening and a disorder structure which is enhanced by the shear deformation and secondly due to the atomic mobility diffusional relaxation process results in densification and thus diffusional ordering of the structure [4]. The deformation is considered inhomogeneous when the temperatures remain well below the T_g (glass transition temperature) and homogeneous deformation occurs when the temperature are above the T_g (glass transition temperature). Strain softening is associated with the accumulation of free volume, thus increasing the STZ volume and the formation and propagation of shear bands that leads to catastrophic failure of the material. This will be dominant when the rate of accumulation of free volume exceeds the rate of annihilation of free volume at low temperatures during deformation this will slow the diffusion process. [1-2, 4]. Thus, this extremely small contribution of the relative volume in a deformed sample is difficult to access by macroscopic methods.

Pan et al [5] was the first to experimentally measure the STZ volume using rate change method by nanoindentation. The measurement was done by using the cooperative shear model (CSM) which was modified by Johnson-Samwer [6]. The smallest individual STZ size in a metallic glass would be approximately 1nm^3 due to shear localization occurring due to collective arrangement of flow defects. The STZs event at low temperature and constant stress first occur erratically of each other due to their immobile nature. They sporadically correlate and self-organize themselves temporally and spatially to form a larger flow zone [1-2]. Therefore, in order to measure the STZ volume of the already strained surface which is of the size of a few microns after micro turning a hypothesis was assumed that the STZ volume of the machined region will decrease as compared to the unmachined region. In the current study, rate jump method using constant strain rate tests through nanoindentation were done. At each strain rate, a series of ten indentations were done. To compare the STZ volume of the micro turned hardened subsurface with the unmachined region, constant strain rate tests were done in both the unmachined and machined subsurface regions. The step wise calculation of shear transformation zone volume using rate jump method by nanoindentation is mentioned below,

1. The estimation of average Hardness 'H' from constant strain rate tests. The hardness values obtained from the tests done at each strain rate tests are averaged and used for the calculation.
2. The strain rate sensitivity (SRS) 'm' can be determined from the average hardness obtained from the strain rate tests and can be given as [3],

$$m = \frac{\partial lnH}{\partial ln\dot{\varepsilon}} \tag{4}$$

where H is the hardness obtained from the constant strain rate experiments and $\dot{\varepsilon}$ is the indentation strain rate applied during the experiment which is expressed as [3],

$$\dot{\varepsilon} = \frac{1}{h}\frac{dh}{dt} = \frac{\dot{h}}{h} = \frac{1}{2}\frac{\dot{P}}{P} \tag{5}$$

From the logarithmic plot of ln(H) Hardness with the ln($\dot{\varepsilon}$) strain rate, the SRS (m) values are obtained, where h is the depth of penetration. A typical plot of strain rate sensitivity 'm' for the unmachined and the machined region is depicted in the Fig. 3.

Fig. 3 illustrates the typical plot of SRS (m) determination for three cases. The linear fit to the curves provides the value of the strain rate sensitivity 'm' for the unmachined and the machined region.

3. The estimation of activation volume V* Activation volume (V*) can be defined as the plastic volume involved in the deformation. The activation volume can be determined from the obtained 'm' values given by the equation [4],

$$V^* = \frac{kT}{m\tau} \approx \frac{3\sqrt{3}kT}{mH} \tag{6}$$

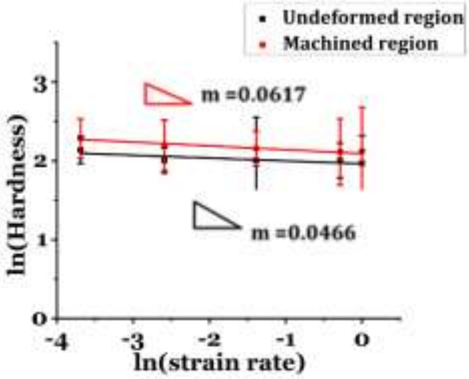

Fig. 3 Typical strain rate sensitivity (SRS) 'm' for 28 m/min and 30 µm/rev

where k represents the Boltzmann constant and T denotes the absolute temperature, respectively. In nanoindentation tests the hardness is equivalent to, H = 3√3τ_y and 'm' is the strain sensitivity obtained (both obtained from constant strain rate test (τ_y=shear strength). The activation volume calculated for the unmachined and the machined region using Eq. (6)

4. The STZ volume (Ω) can be thus calculated from the co-operative shear model equation of STZs modified by Johnson-Samwer [6]. Thus, the Johnson- Samwer equation can be used to estimate STZ volume from the activation volume V* by the equation given as,

$$\Omega = \frac{\tau_0/G_0}{6R'\gamma_C^2\xi\left(1-{}^{\tau_T}/_{\tau_0}\right)^{1/2}}V^* \tag{7}$$

where V* represents the activation volume, R' is a constant which is equivalent to 1/3, τ_T and τ_0 are threshold shear resistances in which $\frac{\tau_0}{G_0} = 0.036$, γ_c is the critical shear strain, the average elastic strain limit γ_c = 0.027, ξ is a correction factor equal to 3 [6]. The value of $\frac{\tau_T}{\tau_0}$ can be evaluated using,

$$\frac{\tau_T}{G} = \gamma_{C0} - \gamma_{C1}\left({}^{T}/_{T_g}\right)^{2/3} \tag{8}$$

In Eq. (8), the shear modulus G has a weak temperature dependency for the metallic glass. γ_{C0}= 0.036 ± 0.002, γ_{C1}= 0.016 ± 0.002 [6]. The STZ volume obtained has been plotted as a function of the cutting parameters to understand the influence of machining parameters (see Fig. 4).

From Fig. 4, it is obvious that the STZ volume is dependent on the machining conditions. The STZ volume for the unmachined region for all the cases of cutting speeds is found to be higher than for the machined region. This result indicates the hardening of the machined surface. The STZ Volume was found to be lower for all the cases of the higher feed of 40 µm/rev for all the cased of cutting speeds. The highest STZ volume for the machined region of 1.36 nm^3 was found to be highest for the cutting speed of 28 m/min. The STZ volume is found to reduce for the machined region for the feed of 40 µm/rev. The STZ volume was saturated below approx. 1 nm^3 for all the cases of machining conditions. This suggests that very few

STZs are generated in the machined region when compared with the unmachined region. The overall reduction of the STZ volume of the machined region could be attributed to the free volume annihilation and thus suggesting the densification of the region as in the case of polymers [10-12]. The minimum STZ volume obtained for all the conditions was 1nm^3 which hints that the shear band formation does not operate beyond a specific embryonic size [7-9]. The free volume sites have been exhausted after machining to generate further STZs and the stress ultimately becomes sufficiently low to drive further generation of STZs thus saturating the STZ volume. Therefore, strain hardening of the machined region could be peculiarly seen.

High cutting speed also signifies the high strain rate process accompanied by high temperature at the tool workpiece interface which results in reduction in the shear transformation zone size. At lower cutting speed (low strain rate), the cutting temperature is low which restricts the creation of free volume and hence resulting in comparatively higher STZ volume was observed.

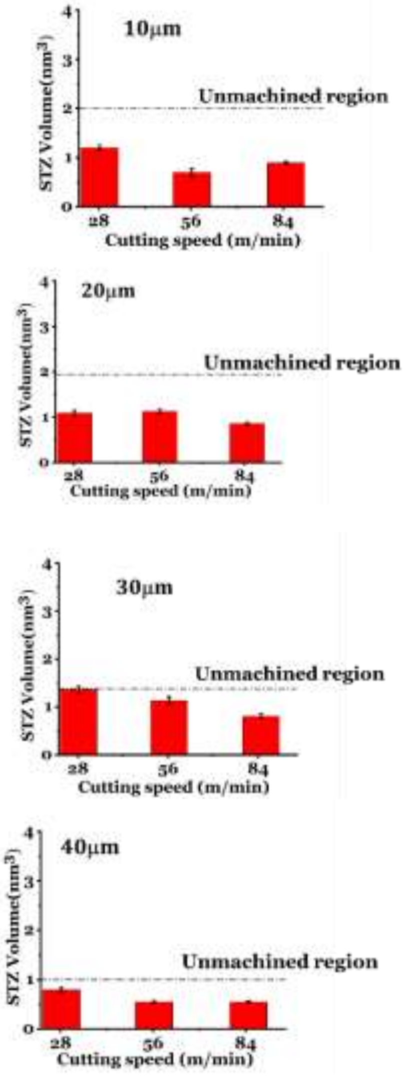

Fig. 4 STZ volume generated at different machining parameters.

4. Conclusions

The following conclusion can be drawn from this work:

1. STZ volume calculated by using Johnson-Samwer equation for the unmachined region and the micro turned region shows lower STZ volume for the micro turned region for all the cases of machining conditions suggesting hardening of the machined sub-surface.
2. The STZ volume is found to be saturated at approx. 1nm^3 for all the investigated machining conditions.
3. Lowest STZ volume is generated for the higher cutting speeds of 84m/min for all feed values except 10μm feed.

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