# Dislocation-like relaxations in cold deformed metallic glasses

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#### Abstract

The results of low temperature (30-300 K) low frequency  $(10^2-10^3 \text{ Hz})$  internal friction investigations of cold deformed metallic glasses (MGs) are summarized. It is shown that inhomogeneous deformation results in a number of internal friction anomalies which are almost identical to those in predeformed crystalline metals. Internal friction of as-cast MGs has no peculiarities. The main features of damping in cold-worked MGs consist in the appearance of (i) large relaxation internal friction peaks analogous to the well-known Hasiguti peaks in crystals and (ii) hysteresis damping which can be suppressed by large predeforming or irradiation. It is argued that inhomogeneous deformation of glassy structures results in the appearance of new dislocation-like defects, not characteristic of the as-cast state, which determine the observed anelastic anomalies.

# 1. Introduction

A study of relaxation processes caused by external actions is one of the most effective ways to investigate physical phenomena in real solids. In particular, this concerns the applications of the internal friction (IF) method. IF measurements can give unique, otherwise unattainable information about the dynamics of the defect structure of solids. The IF method is employed actively for investigations of physical processes which occur in metallic glasses (MGs) during heat treatment. However, the high potentialities of this method as applied to MG plastic flow behaviour remain unused. Several years ago we began to study inhomogeneous (*i.e.* with slip band formation) MG plastic flow by means of IF measurements. It was found that inhomogeneous deformation can result in sharp changes in anelastic properties at  $T \leq 300$  K. The character of these changes is close to, if not identical with, those in crystalline metals caused by dislocation motion in external alternate stress fields. The results obtained are summarized in the present paper. A part of these results is considered in refs. 1-7.

#### 2. Experimental details

MGs (of metal-metal and metal-metalloid type) produced by the usual melt spinning technique as ribbons (30-50  $\mu$ m thick) were used for the investigations. Xray diffraction and transmission electron microscopy were performed to ensure that the ribbons were entirely amorphous. The samples were prepared by cutting off the ribbon into strips (5–10 mm in length, 1–3 mm wide) with the help of a special cutting-out press. IF was measured using the vibrating reed technique. The experimental apparatus permitted determination of the strain tensile amplitude  $\epsilon_0$  during flexural vibrations. The corresponding details and error limits are considered in ref. 4. Predeforming was performed at room temperature by cold rolling or forging. The prestrain value  $\epsilon_p$  was estimated as the average relative thickness decrease.

#### 3. Results

## 3.1. Internal friction in as-cast and deformed state

Generally, after careful gripping IF in the range 30  $K \le T \le 300$  K has no peculiarities and weakly increases with temperature rise. Preliminary inhomogeneous deformation results in a number of IF anomalies. The main anomaly consists in the appearance of a high  $(Q_{\text{max}}^{-1} \text{ can reach } 5 \times 10^{-2})$  IF relaxation peak with a corresponding modulus deficiency of about 10%-15%. The IF peak temperature for the MGs investigated (with crystallization temperatures of 700–900 K) is in the range 220-280 K. The shape, height and temperature of the peak are quite well reproducible on repeated temperature scans of the same sample at fixed  $\epsilon_0$ ,  $\epsilon_p$ and frequency f. However, for various samples of the same MG predeformed by the same quantity, the peak parameters can differ significantly. More often one can observe an asymmetric IF peak but symmetric wide peaks or several (two or three) closely located peaks are also possible. The prestrain value necessary for the peak formation depends on the type of predeformation. In the case of rolling the peak appears after prestraining by several per cent (2%-6%) and in the case of forging a few tens of a per cent are needed. This is illustrated by curves 1,1' and 2,2' in Fig. 1(a) which show temperature dependences of IF and normalized elastic modulus in the as-cast and 3% rolled Ni<sub>60</sub>Nb<sub>40</sub> MG.

It should be emphasized also that in some cases (dependent on MG chemical composition or preannealing) low temperature IF can be extremely sensitive to small plastic deformations and the IF peak is observed even after very careful gripping. However, the employment of special measures allows us to reveal its deformation origin [4, 5].

# 3.2. Influence of deformation amplitude and frequency

The character of IF temperature dependences is a strong function of deformation amplitude and frequency. An increase in  $\epsilon_0$  at fixed f and  $\epsilon_p$  results in an IF peak height increase and in a peak shift by 25–50 K towards low temperatures on an increase in  $\epsilon_0$  by an order of magnitude (curves 2–4 in Fig. 1(a)). In addition, an increase in  $\epsilon_0$  leads to a significant rise of damping at  $T \leq 120-150$  K. IF temperature dependences at various frequencies and fixed  $\epsilon_0$ ,  $\epsilon_p$  which illustrate the characteristic behaviour are shown in Fig. 2(a). The



Fig. 1. (a) Temperature dependences of IF (curves 1–5) and normalized elastic modulus (curves 1'–5') of Ni<sub>60</sub>Nb<sub>40</sub> MG in the as-cast state (curves 1, 1',  $\epsilon_0 = 1 \times 10^{-5}$ ) and after cold rolling by 3% (curves 2, 2',  $\epsilon_0 = 7 \times 10^{-6}$ ; curves 3,3',  $\epsilon_0 = 3 \times 10^{-5}$ ; curves 4,4',  $\epsilon_0 = 5 \times 10^{-5}$ ) and 12% (5,5',  $\epsilon_0 = 2 \times 10^{-5}$ ) ( $f \approx 400-450$  Hz). (b) Influence of 2 MeV electron irradiation on the IF temperature dependence of an Ni<sub>60</sub>Nb<sub>40</sub> sample rolled by 2.5%: curve 1, after rolling; curve 2, after irradiation of the same sample by a fluence of  $1 \times 10^{18}$  cm<sup>-2</sup>.



Fig. 2. (a) IF temperature dependences of Ni<sub>60</sub>Nb<sub>40</sub> MG rolled by 3% at various frequencies: curve 1, 308 Hz; curve 2, 703 Hz; curve 3, 1440 Hz ( $\epsilon_0 = 2.5 \times 10^{-5}$ ). (b) IF temperature dependences of Ni<sub>60</sub>Nb<sub>40</sub> MG rolled by 2.5% (curve 1), annealed after that at T = 523 K for 15 min (curve 2) and rolled again by 1% (curve 3) ( $\epsilon_0 = 2 \times 10^{-5}$ ,  $f \approx 300$  Hz).

peak shifts to higher temperatures and increases in height with elevation of frequency. Typical values of the activation parameters determined by the frequency shift method are as follows: activation energy  $U \approx 0.5$ eV and pre-exponential factor  $\tau_0^{-1} \approx 10^{13} \text{ s}^{-1}$ . It should be noted also that a frequency rise results in no change in the damping at  $T \leq 120-150$  K. Therefore, the IF in deformed samples has a compound character: at  $T \leq 120-150$  K IF is mainly hysteresis and at higher temperatures hysteresis damping is superimposed by a relaxation IF peak.

It must be especially emphasized that IF in as-cast samples is practically amplitude independent. Inhomogeneous deformation always results in a strong IF amplitude dependence and IF hysteresis (at high  $\epsilon_0$ ) as exemplified by Fig. 3 where the IF amplitude dependences of Pd<sub>77.5</sub>Cu<sub>6</sub>Si<sub>16.5</sub> MG at room temperature in the as-cast and predeformed states are shown. It is seen (curves 2–5) that the character of the IF amplitude dependence in the deformed amorphous state is close to that in the crystallized state (obtained by heat treatment of the same specimen). The difference consists in the absence of amplitude-independent damping in the deformed MG even at the very low (down to  $3 \times 10^{-7}$ ) deformation amplitudes used.

# 3.3. Influence of predeforming magnitude, irradiation and heat treatment

Large prestraining results in a lowering IF peak height, a modulus deficiency decrease and a diminishing



Fig. 3. IF amplitude dependence of  $Pd_{77.5}Cu_6Si_{16.5}$  MG at room temperature in the as-cast state (curve 1), after cold forging by 3% (curve 2), 28% (curve 3), 63% (curve 4) and in the crystallized state (curve 5) ( $f \approx 300$  Hz).

IF amplitude dependence. In heavily predeformed samples IF temperature dependences are close to those in the as-cast state (see curves 5,5' in Fig. 1(a)). 2 MeV electron irradiation by a  $10^{18}$ - $10^{19}$  cm<sup>-2</sup> dose suppresses the peak completely (Fig. 1(b)). Heat treatment at modest temperatures  $T \approx 500-600$  K (far from the crystallization starting temperature) during a few tens of minutes results in complete peak disappearance also. Repeated predeformation restores the peak (Fig. 2(b)). It must be pointed out that heat treatment must be carried out without withdrawal of the sample from the clamp. In the opposite case, because of the uncontrolled straining in the course of regripping, one can draw an erroneous conclusion about the stability of the peak within the amorphous state.

# 3.4. The degree of generality of the obtained results

We have studied deformation-induced IF peaks in the MGs  $Pd_{77,5}Cu_6Si_{16.5}$ ,  $Ni_{78}Si_8B_{14}$ ,  $Cu_{50}Ti_{50}$  and  $Ni_{60}Nb_{40}$  in detail [1–7]. Analogous peaks were observed in several MGs based on Fe–Ni and Co. One can believe that the appearance of low temperature IF peaks after inhomogeneous deformation is characteristic of MGs.

# 4. Discussion

The determined sharp change in MG anelastic properties after inhomogeneous deformation indicates a significant change in internal defect structure. One can raise the question of the character of defects responsible for the observed anomalies. We consider that the obtained results corroborate the dislocation concept [6, 8–13] of MG inhomogeneous flow.

The first point for this statement follows from the fact of the appearance of hysteresis damping as a result of deformation. As is generally known [14], hysteresis damping is a typical dislocation effect and is due to a breakaway of dislocations from point obstacles. In nondislocated crystals (whiskers) hysteresis damping is absent [15]. Therefore, one can suppose that the IF amplitude dependence in deformed MGs is conditioned by a breakaway of dislocation-like defects created by predeformation from point anchor centres such as fluctuations of density and/or chemical composition. The similarity of IF amplitude dependences in deformed amorphous and crystalline states (Fig. 3) seems to be definitive in this sense. If one supposes the possibility of an interpretation of hysteresis damping as a result of point-like defect motion, the reason for the absence of this damping in the as-cast state which is definitely characterized by a considerable density of such defects becomes unclear.

The second argument for the dislocation origin of the observed IF anomalies results from the possibility of their suppression by high plastic deformation or irradiation. An analogous situation occurs in the case of crystalline metals [14] and this appears to be one of the most striking pieces of evidence for their dislocation nature. Locking of dislocation-like motion due to either internal stress increase at high prestrain or radiation-induced defects seems to be a probable reason for the disappearance of the considered anomalies in MGs.

The next point for the concept under consideration originates from the determined sufficient lowering of the IF peak temperature with deformation amplitude increase. Such a dependence is not characteristic of point defect relaxation and indicates a high activation volume V of the process responsible for the IF peak. It should be pointed out that such a behaviour is observed in the case of dislocation relaxations in crystals [16]. The activation energy of the relaxation process can be expressed as  $U_{\text{eff}} = U_0 - \sigma V$ , where  $U_0 = \text{constant}$ and  $\sigma$  is the applied shear stress. If the IF measurements are carried out at two different stress amplitudes  $\sigma_1$ and  $\sigma_2$  ( $\sigma_1 < \sigma_2$ ), then the corresponding IF peak temperatures are  $T_1$  and  $T_2$  ( $T_1 > T_2$ ). It is easy to show that  $V = U_0(T_1 - T_2)/(T_1\sigma_2 - T_2\sigma_1)$ . The value of  $\sigma$  can be estimated from Hooke's law:  $\sigma = \epsilon_0 E / \sqrt{3}$ , where  $\epsilon_0$  is the tensile strain amplitude and E is the Young's modulus. Taking for the Ni<sub>60</sub>Nb<sub>40</sub> MG E = 109 GPa [13],  $\epsilon_{01} = 7 \times 10^{-6}$ ,  $\epsilon_{02} = 5 \times 10^{-5}$ ,  $T_1 = 270$  K,  $T_2 = 235$  K (see curves 2, 4 in Fig. 1(a)) and  $U_0 = 0.5$  eV, we obtain V = 3.8 nm<sup>3</sup>. In the units of mean interatomic distance this value is approximately equal to 210. Such a large value of the activation volume contradicts the assumption that point-like defects are responsible for flow but appears to be an argument for a dislocation-like deformation mechanism.

We suppose deformation-induced IF peaks in MGs to be an analogue of the well-known Hasiguti peaks in predeformed crystalline metals. Such a conclusion results from their deformation origin, from their disappearance after large prestraining or irradiation and from their high activation volume. In addition, as determined for the  $Ni_{78}Si_8B_{14}$  MG [5] the IF peak height during room temperature aging increases at first and decreases after prolonged (several months) exposure. Such a behaviour is known for the Hasiguti peaks [14] and indicates participation of both dislocations and point defects in the relaxation process.

The obtained results indicate that inhomogeneous deformation of MGs results in formation of new dislocation-like defects, not characteristic of the as-cast state, which are responsible for the observed IF anomalies.

#### Acknowledgment

The author is grateful to the ICIFUAS-10 Organizing Committee for the financial support for participation in the Conference.

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