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INTERNAL FRICTION PEAKS IN AMORPHOUS Pd$_{80}$Si$_{20}$ ALLOYS

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Abstract.- Two internal friction peaks were observed in amorphous Pd$_{80}$Si$_{20}$ alloys with a conventional torsion pendulum in ascending temperatures. The peak temperature of Peak I (~390°C) was found to be independent of the frequency used but very sensitive to the heating rate of the specimen. The height of this peak increased with the decrease of frequency. The peak temperature of Peak II (546-562°C) changed with frequency as well as with heating rate. Both peaks disappeared during cooling from 600°C to room temperature and they did not reappear when the same specimen was again heated up to 600°C. Differential scanning calorimetric measurements showed a single exothermal peak at 400°C and from the shape of this peak the temperature of crystallization may be defined at 392°C which corresponds to the peak temperature of the internal friction Peak I. The phase transition in the temperature range of Peak II is 'athermal' and its behaviour and mechanism are more complicated.

1. Experimental.- Internal friction of various metallic glass specimens has been much investigated lately but mostly with vibrating reed technique /1-7/. Soshiroda et al. /5/ carried out internal friction measurements on Pd$_{80}$Si$_{20}$ glass with a torsion pendulum at about 0.5 Hz. They found that the zero point on the scale of the optical lever system used in internal friction measurements drifted very rapidly when the specimen was heated to above 350°C, so it was impossible for them to take measurements at temperatures above 350°C. In our present study of the internal friction of amorphous Pd$_{80}$Si$_{20}$ alloys, we used a conventional torsion pendulum of which the total tensile load on the specimen was about 0.4-0.75 Kgmm$^{-2}$. For the frequency range of 0.1-1.0 Hz, the maximum torsion angle on the surface of the specimen was about 10^{-2} radian. Damping curves were obtained from the change of an induced electromotive force in a coil which was fixed onto the lower end of the pendulum. This coil was in a permanent magnetic field and its equilibrium position was recorded with an automa-
tic balance recorder. Fig. 1 shows the linear relationship between the amplitude from the recorder and that obtained simultaneously with an optical lever system. Internal friction measurements were carried out in air and Fig. 2 shows the temperature as a function of time during heating and cooling of the specimen under similar experimental conditions used. Specimens from two different sources were studied and results obtained were not very different. The specimen dimensions were: (1) width, 1.38-2.8 mm; thickness, 60-80 μm; gauge length, 20-28 mm and (2) width, 1.5-2.0 mm; thickness, 80 μm; gauge length, 14-18 mm.

Differential scanning calorimetric measurements were carried out in a Perkin Elmer DSC II. A typical non-isothermal DSC curve of Pd_{80-Si_{20}} is shown on figure 3.

2. Results and discussion.- Fig. 4 shows the results of internal friction measurements, frequency at room temperature was 0.428 Hz. Peak I appeared at 392 ± 1°C and this temperature was found to be independent of the frequency used. The height of this peak increased with a decrease of the frequency of vibration (Fig. 5). The peak temperature was found to be very sensitive to the heating rate used. Peak II appeared around 546-562°C and its position varied with both the frequency and the heating rate. Since the data was somewhat scattered we had to use the 5-point-simulation technique to obtain the peak temperature of the Q'-T curve.

The two peaks appeared only when the specimen was heated to the respective peak temperatures for the first time. After heating to 600°C no peaks were observed during cooling or when the same specimen was reheated. Soshiroda et al. had an amorphous Pd-Si specimen heated to below 380°C and cooled and reheated and they found that the internal friction was going up as before. In our case, we had the specimen heated just beyond the region of Peak I (390°C) and we did not find the peak to appear on cooling, as shown on Fig. 6. When the specimen was heated again to 600°C, the 390°C peak did not reappear but the Peak II was observed. From the behaviour of the internal friction observed we may discuss the internal friction together with the differential scanning calorimetric results of amorphous Pd_{80-Si_{20}} in four temperature intervals:

1) Room temperature — 150°C, internal friction values in this region are about the same for the specimen during either heating or cooling and we may take it as a background.

2) 150 — 350°C, internal friction increases steadily during heating. If the temperature of the specimen was heated to below 350°C, the increase of internal friction with temperature in this range can be re-
Fig. 1: $\Delta \lambda / \lambda$ vs. A

$\lambda$: distance between the mirror on the pendulum and the lamp scale

$\Delta \lambda$: readings on the lamp scale

A: amplitude readings from the automatic recorder

Fig. 2: Temperature vs. time during heating and cooling of the specimen

Fig. 3: A typical non-isothermal DSC curve of Pd$_{80}$Si$_{20}$

(in Perkin Elmer DSC II)
**Fig. 4**: Pd$_{80}$Si$_{20}$ internal friction measurements during heating and cooling (frequency=0.428 Hz at room temperature)

**Fig. 5**: Internal friction Peak I (~390°C) as a function of temperature at different frequencies

**Fig. 6**: Internal friction of Pd$_{80}$Si$_{20}$ glass specimen during heating from room temperature to 400°C and cooling back to 100°C
peated for the same specimen. But when the temperature was lowered after internal friction measurements having been carried out beyond 390°C, Peak I will not appear and the internal friction of this metastable phase appeared much lower than that of the amorphous state. On the DSC curve, Fig. 3, a slight shift of the scanning line in the endothermic direction is detected at a temperature around 300°C. It is obvious then, that a structure change due to the collective movement of atoms in Pd-Si glass in this temperature range is evident from both internal friction and specific heat measurements.

3) 350—400°C, a drastic increase of internal friction occurred in a narrow range of temperature around 390°C, an internal friction Peak I appeared at 392±1°C. This peak is very narrow and sharp. In Fig. 3, a more pronounced shift of the DSC curve in the endothermic direction took place around 360°C. The temperature at this break has been arbitrarily defined as $T_g$ — the glass transition temperature. The DSC curve then turns up at around 380°C in the exothermic direction abruptly and a large DSC peak is observed at 400°C. From the shape of this peak we define the temperature of crystallization for the specimen at this stage to be 392°C. Masumoto and Maddin examined the transformation of Pd$_{80}$Si$_{20}$ glass with X-ray and electron diffraction, electrical resistivity and electron microscopy and concluded that a metastable phase (II) appear in the temperature range 350—550°C, 8/8/. We believe then, this observed internal friction Peak I is a combined result of the large increase of internal friction of the amorphous alloy with temperature around $T_g$ and the onset of a metastable crystalline phase which has a much lower internal friction value at this temperature. The large exothermic peak implies that a large amount of heat is associated with the transition of the amorphous matrix to a metastable phase with a distorted lattice. The position and the shape of this internal friction peak will depend on among other factors the temperature difference between $T_g$ and $T_x$ — the temperature of crystallization of metallic glasses which is known to be sensitive to the heating rate of the specimen. The sensitivity of this internal friction peak temperature to the heating rate used is then a direct consequence.

4) 400—600°C, a broad internal friction peak is observed around 550°C. This peak was very sensitive to both frequency of vibration of the pendulum and heating rate of the furnace. On the DSC trace, we find the slightly sloping base line on the high temperature side of the 400°C exothermic peak continued practically straight from that of the low temperature side of that peak. This range of temperature corresponds to the temperature for the occurrence of a transition of Pd—Si alloy from a metastable phase to a more stable phase as suggested by
Masumoto and Maddin /8/. Since very little thermal change is detected in this range of the temperature in the differential scanning calorimeter, it is suggested that the phase transition of Pd$_{80}$Si$_{20}$ from a metastable phase to a stable phase is athermal. But to understand the mechanism of this internal friction peak II will need more systematic study.

To study the dynamic mechanical properties of amorphous metallic alloys is important in the understanding of the structure and structural stability of these important and unique materials. This preliminary work done on Pd$_{80}$Si$_{20}$ amorphous alloy, one of the much studied metallic glasses, shows that the internal friction technique can be very well used in this field especially if studied together with the differential scanning calorimetric measurements.

References