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Development of the torsion pendulum and early research on grain boundary relaxation and the cold-work internal friction peak*

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Abstract

This paper gives a historical review of the early research on anelasticity during the author's stay at Chicago (1945–1949). Research topics suggested initially by Professor C. Zener were the study of the mechanical properties of grain boundaries and of slip bands in metals by anelastic measurements. The torsion pendulum and the moving coil twisting device were developed for this purpose, so that internal friction, dynamics modulus defect, creep under constant stress and stress relaxation at a constant strain could be measured with the same specimen. The grain boundary peak (*vs.* temperature) was observed for the first time. A "disordered atom groups" model for high-angle grain boundaries was suggested. Similar internal friction peaks associated with slip band relaxation were observed in partially recrystallized cold-worked metals. Anomalous internal friction peaks were discovered in cold-worked and partially annealed Al–Cu solid solutions.

1. Introduction

Late in 1945, the University of Chicago established the Institute for the Study of Metals for physicists, metallurgists and physical chemists. Dr. Cyril S. Smith, metallurgist, then at Los Alamos, was appointed director of the institute and Professor Clarence Zener, then at Watertown Arsenal, was invited by Smith to assist in the initial organization of the institute. After completion of my PhD thesis on spectroscopic research at the University of California, Berkeley in October 1943, I was on the staff of MIT, working in the Spectroscopy Laboratory on a Manhattan District Project on the spectrochemical analysis of uranium and its compounds and in the Radiation Laboratory on discharge research for the transmit–receive switch of long-range radar.

In September 1945, Professor Zener came to the Radiation Laboratory to recruit scientists for the new institute and gave a lecture entitled "Relaxation Spectrum of Metals". I attended this lecture with the understanding that it would be concerned with optical spectroscopy. Although this lecture had nothing to do with optical spectroscopy, I was impressed by Zener's view that the anelastic (acoustic) relaxation spectrum of metals and internal friction had become an important tool in the study of the structure and behavior of metals, and it seemed likely that this research field would come to occupy a position in solid state studies comparable with that posed by the optical spectrum in the theory of atomic and molecular structure. Thus, after Zener's lecture, I expressed my wish to join his new institute and to work on research problems on acoustic relaxation and internal friction.

2. Four years at Chicago – 1945 to 1949

I moved from Cambridge, Massachusetts to Chicago and joined the Institute for the Study of Metals on November 1945, about 1 month after Zener who moved from Watertown to Chicago. Dr. Smith, director of the institute, came in the spring of 1946 together with Professor Charles S. Barrett from the Carnegie Institute of Technology, Professor Earl A. Long from Los Alamos, and some others including Dr. A.W. Lawson and Dr. J.E. Burke.

The institute started its work in the temporary quarters under the stand of the Chicago Stadium, near the site of the ½ W chain-reaction atomic pile constructed by Professor E. Fermi's group. Professor Zener, head of the physics section of the institute, suggested that I concentrated on the mechanical properties of grain boundaries and slip bands in metals. He proposed that the grain boundaries and slip bands should exhibit viscous behavior, and should give rise to relaxation effects and internal friction peaks.

In 1946, Zener had four research associates working on the mechanical properties of metals. The others were Francis T. Worrel, Henry E. Warren and A.W. McReynolds. However, all of them left after 1 year.

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*Invited paper.

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In 1947, L.J. Dijkstra came from the group of Snoek at Philips Eindhoven, Holland, to work in the institute for about 1 year to continue his work on Snoek's internal friction peak. Later in 1948, Charles Wert joined the institute and began his work on the accurate determination of the activation energy associated with Snoek's peak in α-Fe, correlating with Zener's theoretical calculation of the diffusion constant in interstitial diffusion.

In 1949, I returned to Beijing. About 1 month before I left Chicago, A.S. Nowick came from Columbia University to join the institute. He took over the research apparatus built in the previous years and my research problems on point-defect relaxation and the cold-work internal friction peak. Just before my departure on November 1949, the institute started to move into the new building.

3. The development of the torsion pendulum

My first research topic at the Chicago Institute was the study of the mechanical properties of grain boundaries. Zener [1] pointed out that numerous phenomena in the mechanical behavior of the grain boundaries in metals may be explained by assuming that, whatever the exact structure of the boundary, the resistance to sliding of one grain over another obeys the laws commonly associated with amorphous materials. The measurement of anelastic effects (caused by a time-dependent, but recoverable, deformation known as anelastic deformation in addition to the purely elastic, time-independent deformation) therefore offers a powerful tool for the study of the mechanical behavior of grain boundaries in metals. Zener and coworkers [2, 3] had measured the variation of the internal friction at acoustic frequencies with temperature and grain size in Zn, α-brass and α-iron and found that the internal friction increases monotonically with temperature. These results are consistent with the hypothesis that grain boundaries behave in a viscous manner and allow the relaxation of stress across them. Zener suggested that the constraining effect of grain edges and corners in a polycrystalline specimen should produce an internal friction peak when the internal friction is plotted against the temperature of measurement. However, such a peak had not been observed before.

It appears that the failure to observe an internal friction peak may have been due to the fact that the frequency of vibration (kilohertz) used for the internal friction measurement was too high, so that the internal friction curve was shifted towards higher temperatures, and therefore the optimum temperature of the peak was greater than the temperature range accessible by the acoustic measurement technique. Thus measurements at lower vibration frequencies were required.

It occurred to me that the well-known torsion pendulum might be used for the measurement of damping (internal friction) at low frequencies. I recollected that, during my college studies at National Tsinghua University, Peking, China, a torsion apparatus was used to determine the shear modulus of metal specimens in the form of rods. On this basis and using the concept of logarithmic decrement which is widely employed in electric measurements, I realized that the torsion pendulum could be used to measure the damping (internal friction) of a specimen in wire form. Despite the concern that the external damping, including the environmental and clamping losses, would be much higher than the damping caused by internal origins, I went on with my trials and success came gradually. Considering that the new institute did not have any facilities for experiment at that time, the success of the development of the torsion pendulum for internal friction measurement was an achievement. The final form of the torsion pendulum is shown schematically in Fig. 1(a) [4].

In order to study the quasi-static counterpart of internal friction and dynamic modulus measurement, I considered that a twisting device might be used to measure the anelastic creep under constant applied

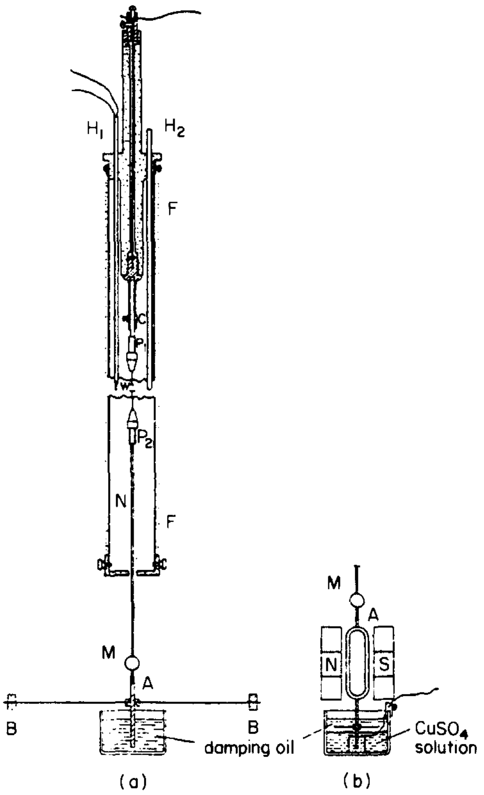

Fig. 1. (a) Apparatus for measuring internal friction and shear modulus of wire specimens. (b) Moving coil arrangement for measuring creep under constant stress and stress relaxation at constant strain of wire specimens.

stress and the stress relaxation under constant strain. This also originated from recollection of an experiment during my college studies involving the measurement of electric current by a wall-type moving coil galvanometer. I thought that the suspension fiber of the galvanometer could be substituted by the test specimen. For this purpose, the torsion bar shown in Fig. 1(a) was unscrewed at A and a moving coil with appropriate damping arrangement was substituted (Fig. 1(b)). The steady current passing through the specimen, which was rigidly connected to a moving coil lying in the uniform magnetic field of a horseshoe permanent magnet, is a measure of the shear stress acting on the test wire and the shear strain can be measured with a lamp and scale optical reflection arrangement. A copper rod was attached to the lower end of the moving coil. This forms one electrode of an electrolytic cell serving as a rotatable current junction. The electrolyte used for the cell is saturated copper sulfate solution and the other electrode of the cell is a copper cylinder surrounding the copper rod.

4. Early work on grain boundary relaxation

4.1. *The discovery of the grain boundary internal friction peak*

Experiments designed to investigate the viscous behavior of grain boundaries at small stress were performed with the torsion apparatus described above. The experimental results confirmed quantitatively Zener's formulation of anelasticity based on Boltzmann's superposition principle. With a frequency of vibration of 0.8 Hz, the variation of the internal friction of polycrystalline aluminum (99.991 wt.%) and "single-crystal" aluminum is shown in Fig. 2 [4]. It is seen that the internal friction reaches a maximum of about 0.09 at about 285 °C for polycrystalline aluminum when the average grain size of the specimen is about 0.3 mm. In the case of the single-crystal specimen, the internal friction is lower over the whole temperature range, and there is definitely no maximum around 285 °C as in polycrystalline aluminum. This conclusion is further confirmed by comparing the internal friction of polycrystalline and single-crystal (prepared using three different methods) aluminum of the same purity [5].

The variation in shear modulus with temperature for the same polycrystalline and single crystal specimens is shown in Fig. 3 [4]. It is seen that the curve is essentially a straight line at low temperatures, and there is a rapid change in curvature around 200 °C for polycrystalline aluminum but not for the single-crystal specimen. The modulus of the single-crystal specimen can be considered as the unrelaxed modulus G_U. $G(T)$ is the modulus of polycrystalline aluminum at temperature T and the ratio $G(T)/G_U$ was plotted as a function of the temperature of measurement. This ratio was found to be about unity below 200 °C, drops suddenly thereafter, and approaches a constant value of about 0.67 at higher temperatures. As a first approximation, we have $G_R/G_U = 0.67$, where G_R is the relaxed modulus. Thus the fractional amount of the total shear stress that can be relaxed across grain boundaries, or the relaxation strength, can be taken as $1 - 0.67 = 0.33$. The constraining effect of the grain edges and corners ensures that the overall stress relaxation is of a limited extent for a fixed overall strain. This G_R/G_U value is close to the value of 0.636 derived by Zener [6] by strain energy considerations.

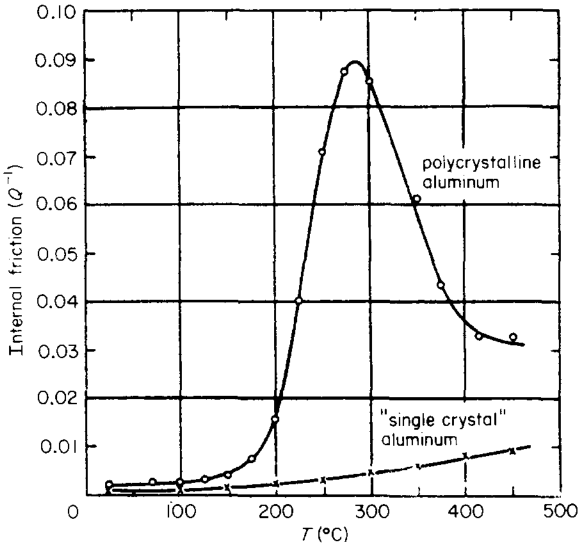

Fig. 2. Variation of internal friction with temperature in polycrystalline and "single-crystal" aluminum (frequency of vibration, 0.8 Hz at room temperature).

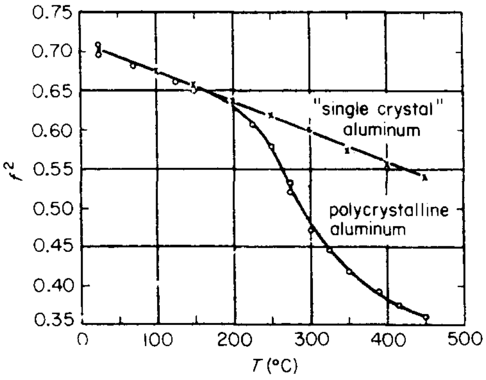

Fig. 3. Variation of square frequency (shear modulus) with temperature in polycrystalline and "single-crystal" aluminum.

The concept of stress relaxation across grain boundaries leads to predictions as to the effect of the frequency f of vibration and grain size (GS) on stress relaxation. Experiments on 99.991 wt.% aluminum showed that this is true; the observed effects are functions of GS, f and T only through a composite parameter $\mathrm{GS}f(\exp(H/kT)$ where H is the activation energy associated with the relaxation process. The value of H was found to be 32 000 cal mol^{-1} or 1.4 eV [7]. The effect of grain size as stated in this parameter only holds for smooth grain boundaries, so that there is no blocking effect to grain boundary sliding except at grain corners.

4.2. Boltzmann's superposition principle and the interrelation between various anelastic effects

In order to generalize Hooke's law so that the time-dependent phenomena can be interpreted, Boltzmann assumed that the fundamental relations between stress and strain are linear in stress and strain and in their time derivatives. The solutions then satisfy the linear superposition principle. This allowed Boltzmann to consider that the strain is also a function of the past history of the stress. Thus the deformation at any instant can be considered as the result of a continuous series of constant forces previously applied. A set of relations was derived accordingly by Zener [8] which holds independent of the magnitude of the observed effects. If these relations can be experimentally verified, then all the effects observed are linear with respect to stress and strain and their time derivatives, and give a self-consistent picture indicating that the grain boundaries behave in a viscous manner. Using these relations derived by Zener, the stress relaxation for different times at 200 °C was calculated in turn from the observed values of the dynamic modulus, internal friction and creep. The results calculated from each type of observation were all in close agreement with the experimentally determined stress relaxation as shown in Fig. 4 [4]. It can be seen that this common stress relaxation curve gives an asymptotic value of the stress function $i_t/i_0 = 0.67$. Thus the relaxation strength is $1-0.67=0.33$.

4.3. Coefficient of viscosity and the structure of grain boundaries

Having demonstrated that the grain boundary behaves in a viscous manner, we are in a position to compute or, at least, to estimate the coefficient of viscosity η and its variation with temperature. It can be shown that [4]

$$\eta(T)=2G(T)\tau_T d/\mathrm{GS} \qquad (1)$$

where $G(T)$ is the shear modulus at the temperature T, τ_T is the relaxation time at T, d is the effective thickness of the grain boundary and GS is the average grain size. Because the forces between atoms in solids are of short range, it is reasonable to consider the thickness of the grain boundary d as of the order of one atomic distance. Thus, for aluminum, we can take $d=4$ Å. Substituting into eqn. (1) the experimental values determined by the anelastic measurements of 99.991 wt.% aluminum described above, the theoretical value of η is 0.18 poise at the melting temperature (659.7 °C); this is close to η of molten aluminum determined experimentally.

A study of the activation energy associated with the viscous sliding along grain boundaries brought about a great advance in our understanding of the structure of the grain boundary. It showed that grain boundary sliding was associated with a diffusion process. Accordingly, the local structure responsible for the viscous sliding along grain boundaries must be imperfections having a local structure which can be considered as separate units. Then the region between such "imperfection units" should be perfect and have a regular structure. Thus the grain boundary structure is heterogeneous and consists of regions of order and disorder. A "disordered atom group" model for high-angle grain boundaries was suggested and the grain boundaries were assumed to consist of many disordered atom groups [9]. The atoms in each group can pass over one another by squeezing the atoms around them. This requires an activation energy which can be supplied by thermal agitation if the temperature is sufficiently high. It can be shown that the coefficient of viscosity associated with this relaxation process is [9]

$$\eta(T)=(kT/V_a)\tau_T d/\mathrm{GS} \qquad (2)$$

where V_a is the activation volume and is independent of stress. The number of "disordered atom groups" in the grain boundary region was assumed to be pro-

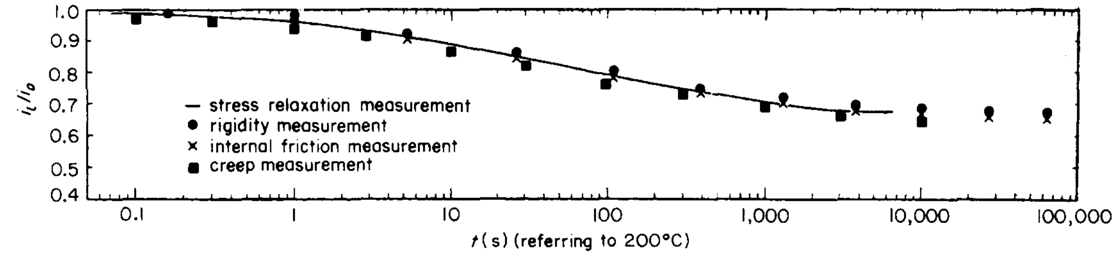

Fig. 4. Stress relaxation across grain boundaries in aluminum at 200 °C as determined by four types of anelastic effect measurement (average grain size, 0.3 mm).

portional to the grain size (GS). Equation (2) can be compared with eqn. (1) derived on the basis of the viscous behavior of grain boundaries.

4.4. Grain boundary relaxation in α-brass and grain boundary structure

For α-brass, internal friction measurements were taken by Zener *et al.* [3] from room temperature up to 450 °C at a frequency between 600 and 900 Hz. The internal friction was found to increase with the temperature of the specimen, but a maximum was never attained. I repeated this experiment with the torsion pendulum for polycrystalline and single-crystal α-brass. It is seen from Fig. 5 [10] that the expected internal friction peak occurs around 415 °C ($f = 0.482$ Hz) in polycrystalline but not in the single-crystal specimen. The activation energy associated with this peak was found to be 41 000 cal mol^{-1} or 1.78 eV, which is close to the activation energy of 41 700 cal mol^{-1} (1.81 eV) [11] for the bulk diffusion of zinc atoms in α-brass (29.08% zinc).

It is seen from Fig. 5 that there is a smaller internal friction peak around 300 °C in both polycrystalline and single-crystal α-brass. It is believed that this peak has the same origin as that observed by Zener [12] in 70–30 α-brass crystal around 420 °C ($f = 620$ Hz). Zener proposed that this peak originated from the stress-induced preferential orientation of pairs of zinc atoms in α-brass [13]. If this picture is correct, the activation energy associated with this peak should be identical with the activation energy for the diffusion of zinc atoms in α-brass. The α-brass crystal used by Zener was furnished by Dr. Cyril S. Smith, then at the American Brass Company, and Zener performed the internal friction measurement by himself. Being a theoretical physicist, this was the only experiment that Zener did and the furnace used was burned out after one measurement run. Thus he obtained only one internal friction curve. The activation energy estimated from the slope of the single internal friction curve plotted against $1/T$ was 33 600 cal mol^{-1} or 1.46 eV, which is much smaller than the value of 41 700 cal mol^{-1} (1.81 eV) for the diffusion of zinc (29.08%) in α-brass [11]. In order to make an accurate determination of the activation energy, I decided to rely on the torsion pendulum for the measurement of internal friction. Polycrystalline wire specimens of α-brass of very large grain size were prepared so that the grain boundary peak was much reduced and the internal friction peak due to preferential reorientation was prominent. The internal friction of this wire was measured with two frequencies of vibration having a ratio of 3.89, from which the activation energy was found to be 40 000 cal mol^{-1} or 1.74 eV [10]. This value agrees well, within experimental error, with the value for the diffusion of zinc in α-brass.

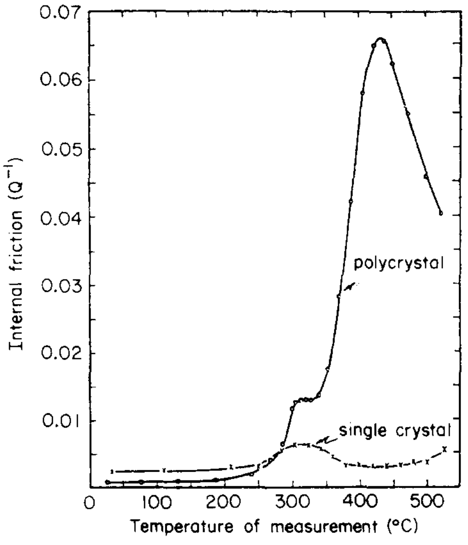

Fig. 5. Variation of internal friction with temperature in polycrystalline and single-crystal α-brass (frequency of vibration, 0.5 Hz).

The activation energy associated with grain boundary relaxation in 70–30 α-brass was found to be 41 000 cal mol^{-1} as reported above. Using the same specimen, the activation energy associated with the preferential orientation of pairs of zinc atoms in α-brass, which is a diffusion phenomenon, is 40 000 cal mol^{-1}. Both values are equal, within experimental error, to the activation energy for the volume diffusion of zinc atoms in α-brass. This shows that the grain boundary relaxation in α-brass, as manifested in the anelastic measurement, is definitely a process of atomic diffusion. A grain boundary model of "disordered atom group" based on this consideration has been described in Section 4.3.

5. Early work on cold-work internal friction peak and slip band relaxation

The second research topic suggested to me by Zener was the mechanical properties of slip bands. Zener proposed that the slip bands should behave in a viscous manner similar to that exhibited by grain boundaries. He was inclined to consider the slip band as a single entity irrespective of the type of structure. Accordingly, there should be an internal friction peak associated with the viscous behavior of slip bands. For this purpose, I started from a study of the internal friction of cold-worked metals and the effect of annealing on this internal friction. The progress of this research was not as straightforward as for grain boundary relaxation.

5.1. Research on the cold-work peak

Historically, Snoek [14] was the first person to observe an internal friction peak near 200 °C for $f = 0.2$ Hz in cold-worked iron containing carbon or nitrogen. He thought that the internal friction was a pure Gorsky damping [15], *i.e.* carbon or nitrogen atoms migrate from regions of positive stress to regions of negative stress in the manner proposed for the first time by Gorsky [15].

In 1948 [16], I began a systematic anelastic study on cold-worked Westinghouse "Puron" (containing as chief impurities 0.040 wt.% oxygen, 0.005 wt.% carbon and 0.004% nitrogen). The internal friction was measured as a function of temperature after the specimen had been annealed at successively higher temperatures. The variation of internal friction with temperature after successive annealings is shown in Fig. 6. There are three internal friction peaks, A, C and E, occurring at temperatures around 20 °C, 225 °C and 490 °C ($f = 0.5$ Hz) respectively. In addition to these peaks, there are three regions, E, D and F, over which the internal friction varies with temperature. Auxiliary experiments showed that peak E is the grain boundary peak in α-iron. It can be seen from Fig. 6 that there is no 20 °C peak (Snoek peak) for the cold-worked specimen and the 225 °C peak disappeared when the specimen was "recrystallized". The 20 °C and 225 °C peaks are complementary, one increases as the other decreases. The 225 °C peak is that first observed by Snoek [14].

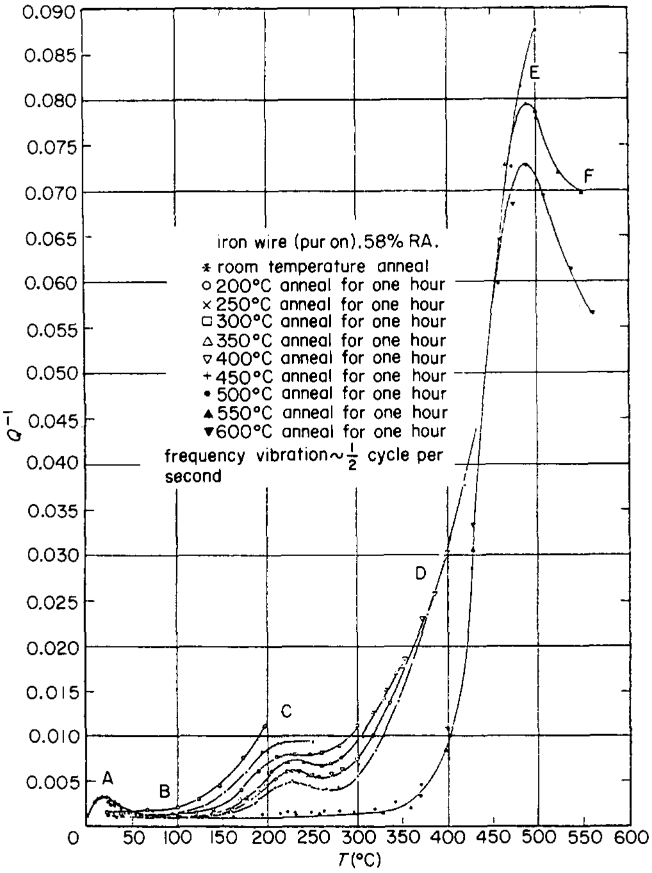

Fig. 6. Variation of the internal friction with the temperature of a cold-worked (58% reduction in area (RA)) iron specimen after successive annealing at various temperatures (in argon) for 1 h. Frequency of vibration, 0.5 Hz.

Subsequent experiments with "Puron" specimens, "purified" but loaded with nitrogen, showed that the conditions for the appearance of this 225 °C peak were that an iron specimen contains a small amount of nitrogen (or carbon) and has been subjected to cold work. The activation energy associated with this peak was found to be 32 000 cal mol^{-1} or 1.4 eV. Accordingly, this peak is associated with the stress-induced diffusion of nitrogen within some "peculiar stress region" produced by cold working (signifying dislocations). The diffusion process involved must be quite different from the interstitial diffusion of nitrogen in a solid solution of iron, because this peak is mutually exclusive with the 20 °C peak, which is caused by the stress-induced interstitial diffusion of nitrogen atoms in annealed iron (Snoek peak). From the experimental data obtained, the distance over which the nitrogen diffuses under the influence of the alternative stress applied during internal friction measurements was estimated to be a few atomic diameters. This showed that the picture of Gorsky damping suggested by Snoek was not adequate.

From the historical review given above, it is evident that the name "cold-work peak" was given to the 225 °C peak which appears in cold-worked specimens merely to differentiate it from the 20 °C peak (Snoek peak) which appears in annealed specimens.

5.2. *Internal friction in partially recrystallized cold-worked metals and relaxation across slip bands*

Cold working can increase the internal friction of metals. If we regard plastic deformation as occurring through the generation and propagation of slip bands, the internal friction may be interpreted as arising from the viscous behavior of these slip bands. Similar to grain boundaries, we can attempt to interpret the structure of partly recovered cold-worked metals in terms of the associated anelastic behavior.

Zener suggested that slip bands are stress-relaxing interfaces which differ from grain boundaries only in that they are irregular, thus relaxation effects are coupled, while grain boundaries are regular. Consequently, the internal friction curve *vs.* temperature of partially recovered cold-worked metals does not yield a peak but seems to rise indefinitely. However, under certain conditions, we cannot exclude the possibility of obtaining some kind of distribution of the slip bands in partially recovered cold-worked metals so that an internal friction peak associated with the viscous behavior of the slip bands can be observed. Such a peak exists in at least two cases [17] (this was not recognized while I was at Chicago). One example is provided by the study of the anelastic behavior of α-iron (Puron). In Fig. 6 [16], a series of internal friction curves of cold-worked (58% reduction in area) iron is shown after successive annealing at various temperatures (1 h). Our present interest is principally in the "relaxation D" shown in the figure. It is seen that there is an abrupt drop in internal friction in region D after the specimen is completely recrystallized (500 °C anneal for 1 h). The difference between the 400 °C anneal curve and the 500 °C anneal curve is shown in Fig. 7 [17]. It is seen that an internal friction peak appears at 400 °C. Another example is the internal friction curves of cold-worked (34% reduction in area) 99.991 wt.% aluminum after annealing at successively higher temperatures. The difference between the 350 °C anneal curve and the 400 °C anneal curve gives an internal friction peak at 225 °C.

The paper entitled, "Structure of cold-worked metals as deduced from anelastic measurements", was published with joint authorship of Professor Zener [18]. The specimen used in the study was 99.991% aluminum which was heavily cold worked but partly recovered by annealing at 250 °C for 2 h. Creep measurements were taken at 200 °C and the total strain at the end of the test (4 h) was nine times the initial elastic strain, yet the creep recovery was essentially complete. We can regard the individual crystals in a cold-worked specimen as containing a network of slip bands, and regard these slip bands as responding to a shear stress in the same viscous manner as the grain boundaries. Accordingly, the localized relaxing regions can be identified with slip bands. However, it appears as if such large recoverable creep would not be caused by a regular network of slip bands; rather we anticipate a very wide distribution in the size of the slip bands, regions between slip bands of one size being interspersed with smaller slip bands. Such a random distribution of slip bands would give rise to a large recoverable creep by "coupled relaxation". The initial relaxation across the smaller slip bands will be relaxed further through the subsequent relaxation across larger slip bands; therefore the total strain will be multiplied repeatedly with the relaxation of successively larger slip bands. The total anelastic strain caused by n groups of slip bands can be represented by the following approximate formula

$$\epsilon(10^{r}t) = (1+\lambda)^{1+2+3+\cdots\cdots+n}\epsilon_0 \tag{3}$$

where ϵ_0 is the elastic strain and λ is a factor representing the anelastic strain over the elastic strain. This formula closely represents the data of the creep curve measured at 200 °C, with the appropriate value of λ being 0.20, which is reasonable.

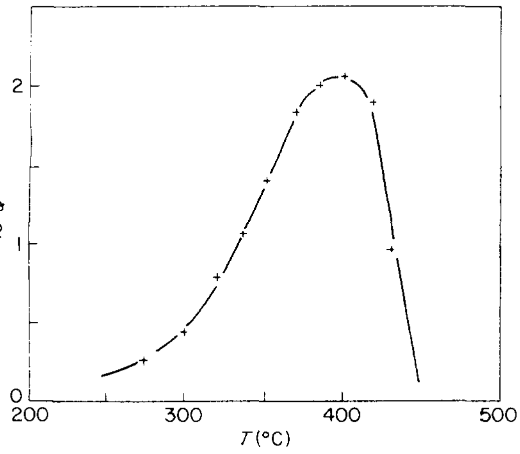

Fig. 7. Internal friction peak associated with the viscous behavior of slip bands in partly recovered cold-worked (58% RA) iron, obtained by subtracting the internal friction of the 500 °C anneal curve from the 400 °C anneal curve shown in Fig. 6.

5.3. *Anomalous internal friction peaks in cold-worked and partially annealed aluminum–copper solid solutions*

Anomalous internal friction peaks were observed in cold-worked aluminum containing 0.5 wt.% copper (partially annealed) in the temperature range −5–125 °C ($f \approx 1$ Hz) as shown in Fig. 8 [19]. At temperatures within the region of the internal friction peak (*vs.* temperature), amplitude–internal friction peaks were obtained when the internal friction was plotted as a function of the strain amplitude (Fig. 9) [20]. Systematic experiments showed that this anomalous internal friction is caused by the presence of copper in cold-worked aluminum. It was not observed in high-purity aluminum under similar conditions. It was eliminated after the

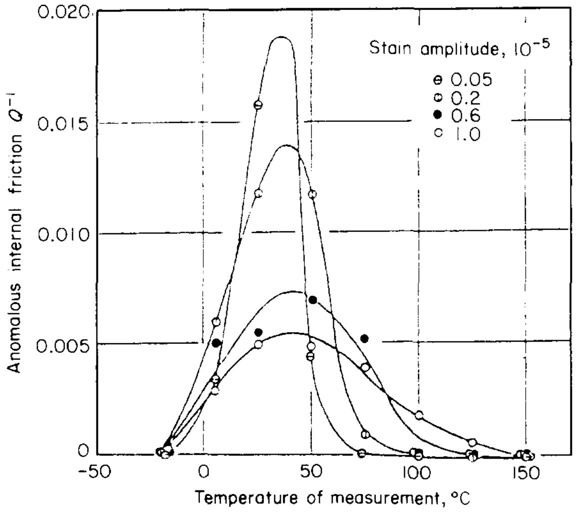

Fig. 8. Temperature dependence of the anomalous (non-linear) internal friction at various strain amplitudes in cold-worked aluminum containing 0.5 wt.% Cu after 1 h annealing at 300 °C.

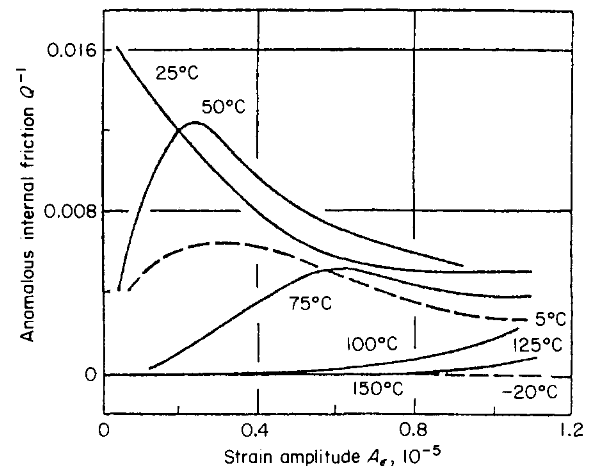

Fig. 9. Amplitude curves for the internal friction in the region of the temperature peaks shown in Fig. 8.

copper-bearing aluminum, originally cold worked, was completely recrystallized. This shows that this peak is related to the presence of dislocations. The aging effect exhibited by the internal friction indicates that the Cu solute atoms interacting with the dislocations are mobile under the experimental conditions. Thus the anomalous effect was interpreted using the concept of the interaction between solute atoms (Cu) and dislocations, whereby solute atoms are trapped by dislocations and, conversely, dislocations are immobilized by an atmosphere of solute atoms.

6. Auxiliary achievements

(1) A vacuum acoustic transverse vibration equipment was constructed for making anelastic measurements at acoustic frequency [21]. The electromagnetic method was initiated for the first time from the comprehension of the earphone principle. In order that this method could also be used for non-ferromagnetic specimens, a thin soft iron disk was attached by Insalute cement to each end of the specimen and an electromagnetic coil, made by winding glass-insulated copper wires around Alnico magnets, was placed immediately beneath each disk. The specimen was supported horizontally at its nodes of vibration with very thin alumel and p-chromel wires; the hot junction of this thermocouple is, thus, the specimen itself. This equipment was used for the study of the grain boundary relaxation in copper and the mechanism of embrittlement of copper by the segregation of bismuth along grain boundaries [21].

(2) Whilst investigating the internal friction peak associated with slip band relaxation, I tried to use specimens with a high melting point and high recrystallization temperature so that the slip bands introduced by cold working could survive the subsequent annealing treatment. Tantalum was used because "high-purity" tantalum was available from the Franstesl Metallurgical Corporation, Chicago. Thus Snoek relaxation in a b.c.c. metal other than α-iron containing interstitial C, N and O was observed for the first time [22].

(3) A simple torsion apparatus was developed for the measurement of extremely high internal friction by determining the angle by which strain lags behind stress in forced cyclic vibration [23]. The measurements of strain and stress were made by two separate moving coil "galvanometers" as described in Fig. 1(b) [4]. In the strain or specimen "galvanometer", the specimen, in wire form, constitutes the suspension fiber of the galvanometer, the current passing through the galvanometer coil is a measure of the shear stress acting on the wire specimen and the deflection of the galvanometer is a measure of the shear strain. The current passing through the coil of this "strain galvanometer" was measured by another moving coil galvanometer with a 0.5 mm piano wire as its suspension fiber. The internal friction of this suspension fiber is very low and the phase lag in this "stress galvanometer" is negligibly small. The deflection of this galvanometer is thus a measure of the applied shear stress.

The cyclic current was generated by driving a sliding contact of a circular potentiometer linearly wound around its entire circumference by a geared-down meter. The period used was 19.9 s. The waveform of the periodic voltage generated by this arrangement is thus a sawtooth and each branch of the sawtooth is linear.

The phase angle between stress and strain was measured by observing the deflections of the strain galvanometer and the stress galvanometer on the same scale. The reading error causes an uncertainty in the value of internal friction of ± 0.005 under good conditions. The highest internal friction measured with this apparatus was 0.55. It allows the measurement of internal friction at a constant given stress or strain amplitude. The stress amplitude and frequency of vibration can easily be varied over a wide range.

7. Concluding remarks

Starting from Zener's suggestion to study the mechanical properties of slip bands as an entity, I turned my attention gradually to the theory of dislocations. Shortly before I left Chicago, the anomalous (nonlinear anelastic) internal friction associated with the interaction between dislocations and solute atoms was observed, and my first report was written during my journey to China and submitted to *Physical Review* from Honolulu. This research topic was continued in China (Beijing, Shenyang and Hefei) cooperating with more than ten young Chinese scientists and graduate students

[24]. In 1979–1980, I visited Stuttgart, Germany by invitation of Professor A. Seeger (Max-Planck-Institut für Metallforschung, Institut für Physik), and in 1980–1981, I visited Villeurbanne, France by invitation of Professor P.F. Gobin (INSA de Lyon), in both cases to carry out cooperative research on dislocation–point defect interaction.

The research on grain boundary relaxation was resumed from 1982 in the newly organized Institute of Solid State Physics, Academia Sinica, Hefei, China. The results on grain boundary relaxation in 99.991 wt.% aluminum, observed in 1947 [4] at Chicago, were reinvestigated, by comparing the internal friction of polycrystalline and single-crystal specimens of the same purity and to ensure that the single-crystal specimen did not contain any fine crystals or bamboo grain boundaries and the specimen had not been cold worked. Later, a conspicuous bamboo boundary internal friction peak was observed simultaneously on one internal friction curve with the conventional fine-grained grain boundary peak in sheet specimens consisting of mixed grains. It was proposed that dislocation substructures exist near the bamboo boundaries, intersect and interact with the bamboo boundaries and are dragged along with the bamboo boundaries during viscous sliding, so that the sliding process is limited by the appearance of an internal friction peak [25]. Very recently, nonlinear anelastic internal friction peaks (*vs.* temperature and strain amplitude) have been observed in bicrystals of aluminum [26].

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