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The α and β Peaks in Cold-Worked Niobium

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Internal-friction measurements (at 1 c/sec) have been carried out over the temperature range from 90 to 290° K, on niobium specimens deformed at room temperature. In the as-cold-worked material, a broad peak at about 115° K (α peak) is observed. The α peak increases with the amount of deformation and decreases with increasing interstitial impurity content. On subsequent annealing, the height of the peak decreases by about 50% over the temperature range from 90 to 140° C, and to negligible values from 250 to 340° C.

As a result of annealing for 2 h at 70° C, a group of peaks (β peaks) occurred at about 200° K. The β peaks are independent of the amount of deformation prior to annealing and the interstitial impurity content. On further annealing, the relaxation strength of the peaks increases with temperature up to about 100° C, remains constant between 100 and 240° C, and subsequently gradually decreases to negligibly low values at about 340° C.

The α peak, and its variation with deformation, impurity content, and annealing, can be accounted for in terms of relaxation mechanisms involving dislocations (i.e. a Bordoni- or Hasiguti-type process observed in fcc metals). This is a generally accepted concept at present. The β peaks, on the other hand, could only be adequately accounted for by relaxation processes involving complexes of deformation-created point defects and interstitial impurities.

1. Introduction

Internal-friction relaxation peaks at sub-zero temperatures in cold-worked niobium have been denoted α and β peaks by Chambers and Schultz [1, 2]. At a frequency of oscillation of 1 c/sec, the α peak occurs at about 100° K and the β peak at about 200° K. The spectrum of internal friction at these temperatures indicates that, in each case, there is more than one relaxation process in operation [2, 3]. In fcc metals, relaxation peaks occurring at sub-zero temperatures are caused by mechanisms involving either pure dislocations (Bordoni peak) or dislocation-point-defect relaxations (Hasiguti peak). The relaxation mechanism of the Bordoni peak is based on the thermally activated motion of dislocation kinks over energy barriers along the Peierls barrier [4]. The Hasiguti mechanism, on the other hand, involves the breaking away, under cyclic stressing, of dislocation lines from their atmospheres of point defects. Before these dislocations have had the opportunity to return to their original positions, the point defects migrate to them and re-pin them. The repeated breaking and re-forming of the pinning points results in a relaxation peak [5].

It is now clear that the α and β peaks in bcc metals cannot be accounted for by the models used to explain the Bordoni and Hasiguti peaks in fcc metals [2]. This is primarily due to the basically different annealing behaviour of the α and β peaks from the Bordoni and Hasiguti peaks respectively. It is, however, currently agreed that the motion of dislocations is directly involved in the relaxation processes causing the α peaks. With regard to the β peaks, Cham-

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bers and Schultz [6] and de Batist [3] propose that these peaks are also due to a relaxation mechanism involving dislocations. On the basis of studies of both cold-worked and irradiated niobium, the present authors have proposed that the β peaks are more likely to be due to relaxations involving point-defect complexes which are not associated with dislocations at all [7, 8].

The purpose of this paper is to present a study of the effect of the degree of cold-work, interstitial impurity content, and post-cold-work annealing on the α and β peaks, as measured using a low-frequency (1 c/sec) internal-friction apparatus, with a view to contributing to the understanding of the relaxation mechanisms causing the β peaks.

2. Material and Experimental Procedure

The material used in this work was the same niobium of commercial purity as that used in the earlier investigations [7, 8]. Methods of preparing the testpieces, gas analysis, and straining were also as described previously. Internal-friction measurements were carried out in the low-frequency (1 c/sec) torsion pendulum apparatus described in a previous paper [8].

3. Results

3.1. The α Peak

A selection of α peaks developed after room-temperature deformation to varying strains is shown in fig. 1. In general, the height of the peak tends to increase with strain, which is in broad agreement with the results of Chambers and Schultz [6]. The peak temperature decreases from 118 to 112° K, as the strain is increased from 0.14 to 4.5%. Chambers and Schultz [6] also noted a slight decrease of peak temperature with increasing strain. However, in their specimens, the peak temperature started to rise again at strains higher than 2%. In view of the indication that impurities in bcc transition metals tend to depress the α peak, the height of the peak has been plotted against the ratio of the strain (ϵ) to the interstitial impurity content plus 1 ($c + 1$) (fig. 2). The four points (marked O) in the figure, representing specimens strained within the fairly narrow range from 3.9 to 4.5%, indicate that the peak in the curve is due to the impurities present and not to the degree of deformation.

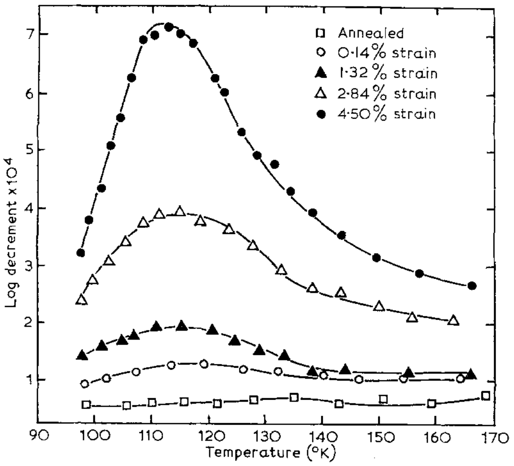

Figure 1 Effect of strain on the α peak in cold-worked niobium.

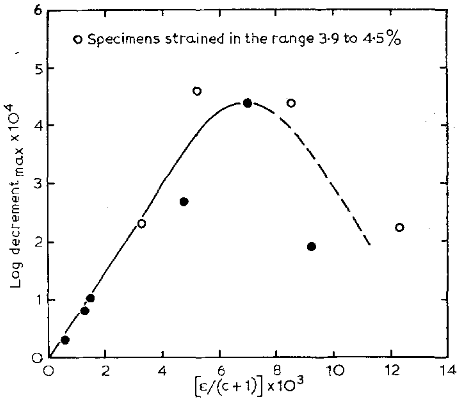

Figure 2 Variation of the height of the α peak with strain (ϵ) and total interstitial impurity content (c).

From the shift of the temperature of the α peak with frequency of oscillation, a lower limit, about 0.3 eV, of the activation energy of the relaxation process was obtained. This value is in good agreement with the range of values, 0.23 to 0.28 eV, determined by other workers [6, 9, 10].

3.2. The Background Damping

The background damping is shown in fig. 3, plotted against $\epsilon/(c + 1)$. Over the range of strain and impurity content investigated, the background damping increases both with increasing strain and with decreasing interstitial impurity content.

3.3. The β Peak

Annealing of room-temperature-deformed speci-

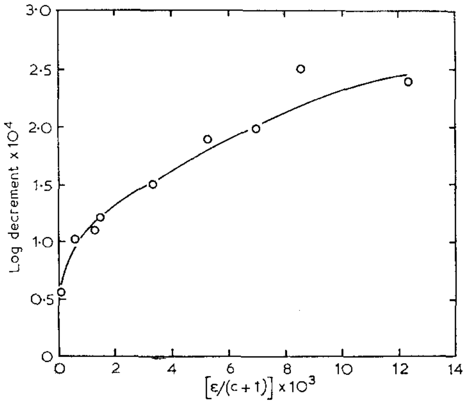

Figure 3 Variation of background damping with strain (ε) and total interstitial impurity content (c).

mens for 2 h at 70° C resulted in the appearance of relaxation peaks in the temperature (200° K) region of the β peak in bcc transition metals [2], whilst the height of the α peak and the magnitude of the background damping remained unaffected. Typical internal-friction curves showing this effect are given in fig. 4. It should be noted, however, that Chambers and Schultz [6] observed well-developed β peaks after room-temperature deformation without the need of annealing at higher temperatures.

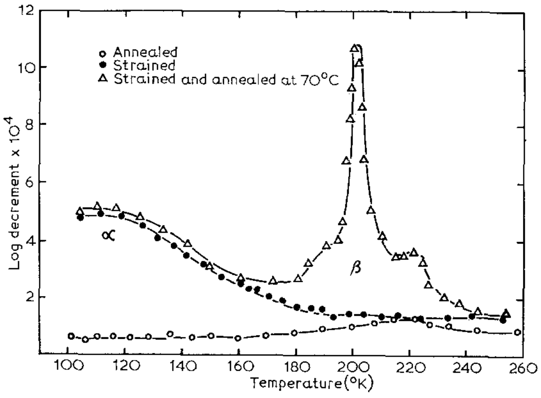

Figure 4 α and β peaks in cold-worked niobium.

The β-peak complex consists of a narrow main peak occurring at about 200° K, and smaller subsidiary peaks occurring at about 190 and 220° K. The subsidiary peaks, in contrast to the main peak, occurred in some, but not all, of the specimens examined. Another unusual feature of the present results, which is not common with those of other workers [3, 6], is that only the main peak of each specimen grew to a significant height on annealing at 70° C. This is particularly in sharp contrast with the group of well-developed peaks occurring over the temperature range from 190 to 250° K, in the same material but neutron-irradiated [8].

No definite relationship has been observed between the height of the main β peak and the degree of strain or the interstitial impurity content (table I). It is of interest to note, however, that no peak exceeded a value of 9 × 10^{-4} of the logarithmic decrement obtained after a strain of only 0.14%

TABLE I Effect of degree of strain and effect of oxygen and nitrogen content on the height of the main β peak.

Strain (%)	Nitrogen content (at. ppm)	Oxygen content (at. ppm)	Total interstitial content (at. ppm)	β-peak height (log dec × 10^4)
0.14	106	145	251	9.1
1.32	378	650	1028	8.9
1.50	153	875	1028	3.6
2.84	100	208	308	8.3
3.90	119	197	316	6.6
3.94	359	852	1211	8.6
4.30	332	492	824	4.6
4.50	252	354	606	4.1
4.82	166	528	694	7.3
5.16	392	701	1093	8.7

An approximate estimation, from the frequency shift of the peak temperature, of the activation energy of the main β peak yielded a value of about 0.6 eV. This value is higher than the 0.46 eV reported by other workers [6, 10]. On the other hand, the lower limit of the activation energy, estimated from the half-width of the peak, was found to be 1.02 eV, which is even higher than the value obtained from frequency shift. The high value of the activation energy indicates that the β peak is much narrower than a Debye relaxation peak.

3.4. Effect of Annealing

Variation of the α and β peaks and the background damping with annealing temperature of a specimen strained to 3.8% is shown in fig. 5. The β peak increases appreciably from zero at room temperature to a logarithmic decrement value of about 13 × 10^{-4} at about 100° C. Over a temperature range from 100 to 240° C, the peak remains practically constant at about this

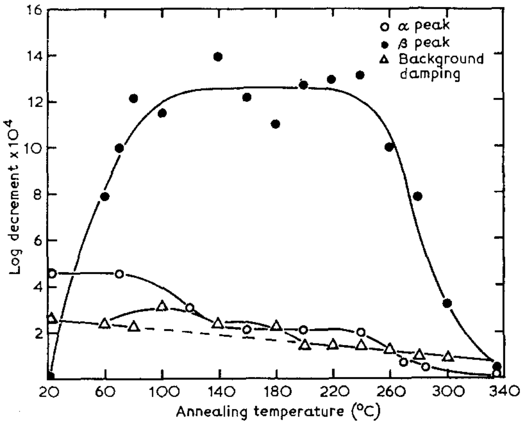

Figure 5 Isochronal annealing curves of the α peak, the main β peak, and background damping of a specimen strained to 3.8%.

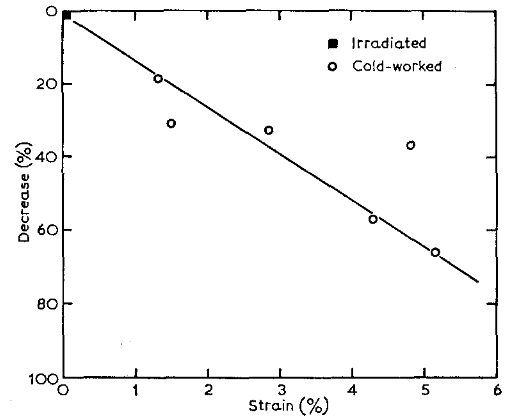

Figure 6 Decrease in height of the main β peak in cold-worked niobium after re-straining to 0.3% and annealing for 20 h at room temperature.

value. At temperatures higher than 240° C, the peak anneals out to a negligibly low value at about 340° C. The height of the α peak, on the other hand, remains unaffected until a temperature of about 90° C is reached, above which it anneals out to approximately half its original value at about 140° C. Thereafter, the peak height remains constant until a temperature of about 250° C is attained, above which it anneals out to zero at about 340° C. The background damping decreases linearly over the whole temperature range with the exception of two slight increases at about 120 and 180° C.

3.5. Effect of Re-straining

In a previous paper [7], it was found that the effect of an additional 0.3% plastic deformation at room temperature on the main β peak was to cause the peak to decrease in height with time at room temperature, so that, after 40 min, the height was reduced to two-thirds and, after 20 h, to one-third of its original value, whilst there was no significant change in the height of the α peak. The same experiment was carried out on specimens which were cold-worked to different amounts of plastic strain and annealed for 2 h at 70° C. Internal friction in all cases was measured 20 h after re-straining. In each specimen, the main β peak decreased in height. The decrease is plotted against the initial pre-strain in fig. 6. The graph includes an equivalent result obtained on an irradiated specimen [8], which represents the case of zero pre-strain. The relationship is linear, with the straight line passing through the origin.

Each of the re-strained specimens was then re-annealed for 2 h at 70° C, and internal friction was measured again. In all but one case, the height of the main peak increased. The percentage increase is plotted against the initial pre-strain in fig. 7. Despite the scatter of these results, it can be seen that the percentage increase in the height of the β peak tends to increase with increasing degree of cold-work.

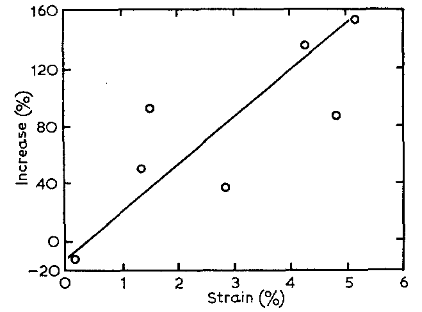

Figure 7 Increase in height of the main β peak on annealing for 2 h at 70° C, following the 0.3% re-straining and room-temperature re-annealing treatment.

The specimen, which was initially strained to 5.16%, annealed at 70° C, re-strained by 0.3%, and re-annealed at 70° C, was again strained by a further 0.3%, and the internal friction was measured after 20 h annealing at room temperature. This time, a decrease of 14% in the height of the peak was observed (table II), which is significantly less than the decrease of 65% observed after the first 0.3% re-strain and 20 h room-temperature annealing. Finally, this speci-

TABLE II Effect of annealing and re-straining by 0.3% on the main β peak of a specimen strained to 5.16%.

Treatment	β-peak height (log dec ×10^4)	Change in height (log dec ×10^4)	Incremental change (%)
1st anneal at 70° C	8.7	—	—
1st re-strain	3.0	−5.7	−66
2nd anneal at 70° C	7.6	+4.6	+153
2nd re-strain	6.6	−1.0	−14
3rd anneal at 70° C	7.1	+0.5	+7

men was re-annealed once more at 70° C for 2 h, and the internal friction was measured. This time, the peak increased in height by only 7%, a negligible amount compared with the 153% increase observed after the second anneal at 70° C (table II).

4. Discussion

In view of the fact that α and β peaks in commercial niobium, whether cold-worked or irradiated, occur over the same temperature range as those described by Chambers [2] in bcc transition metals, it may be assumed that these peaks are caused by the same mechanisms. The ensuing discussion will, therefore, follow the same lines as in the case of irradiated niobium [8], i.e. an attempt will be made to interpret critically these peaks in terms of mechanisms that are most likely to operate at the temperatures at which these peaks occur. The mechanisms to be considered are: (i) a dislocation relaxation (Bordoni type); (ii) a dislocation-point-defect relaxation (Hasiguti type); and (iii) a point-defect or point-defect-complex relaxation.

4.1. The α Peak

The α peak cannot be accounted for in terms of relaxation mechanisms involving point defects, for the following reasons. Firstly, the height of the α peak in the cold-worked specimens should have been lower than it was observed to be in the same material after irradiation [8], the main reason being that irradiation produces many more point defects than plastic deformation. Secondly, for a given degree of deformation, the peak should be continuously reduced by an increasing amount of interstitial impurities, owing to the increased number of point defects being trapped by the impurities. The height of the peak actually goes through a maximum when plotted against total interstitial impurity content (open circles in fig. 2). Thirdly, because of trapping, the height of the peak should commence to decrease as soon as the annealing temperature is increased above room temperature, and not wait until 90° C is reached, when oxygen atoms are mobile [11].

Bordoni mechanism The fact that the height of the α peak increases with increasing degree of strain (fig. 1), without any heat-treatment at temperatures above the cold-work temperature, indicates that the relaxation mechanism causing the peak involves dislocations. This is in agreement with a generally accepted concept at present [2, 3, 8]. As the interstitial impurity content increases, the number of dislocations produced for a given strain also increases [12, 13], resulting in a greater number of dislocation relaxations and, consequently, a higher α peak. However, as the number of dislocations produced by a given strain is increased, the average loop length of the dislocations decreases owing to dislocation intersections. Seeger's model [4] for the Bordoni peak in fcc metals predicts a ΛL^3 dependence of the relaxation strength, where Λ is the dislocation density, and L is the average loop length. Therefore, above a certain dislocation density, the L^3 term will predominate over the Λ term. This implies a reduction in the relaxation strength. The decrease in the height of the α peak at the higher impurity concentrations, which would produce greater densities of dislocations, can, therefore, be accounted for by the dislocation relaxation model.

The decrease in the height of the α peak on annealing in the temperature range from 90 to 140° C is probably due to the migration of vacancies and/or oxygen atoms to dislocation loops, reducing the relaxation strength. The higher temperature range from 250 to 340° C, in which the α peak shows a further decrease in height, is the range in which nitrogen atoms are expected to be mobile. Thus, this decrease could be attributed to the migration of nitrogen atoms to the dislocations. The lack of appreciable change in the peak height after an additional 0.3% strain, which is to be expected from the plot of the height of the α peak versus pre-strain (fig. 2), will be due to the pinning of the relatively few long loops produced by the small strain by point defects able to migrate at room temperature.

Hasiguti mechanism The results can also be satisfactorily explained by the dislocation-point-defect hypothesis (Hasiguti type). For example, the annealing stages of the peak, 90 to 140° C and 250 to 340° C, could be attributed to the migration of interstitial impurities, oxygen and nitrogen respectively, to dislocations forming strong pinning points, and therefore reducing the number of dislocation loops that can take part in the relaxations. The negligible change in the height of the peak after the 0.3% re-strain could also be accounted for by the migration of point defects at room temperature to the freshly unpinned loops.

4.2. The β Peaks

The results of the present work show that the α and β peaks in cold-worked niobium exhibit a markedly different behaviour (table III). This lack of similarity and correlation between the behaviour of the two types of peaks indicates that the peaks are caused by differing relaxation mechanisms. Since, in the literature [2, 3, 8], there is general agreement, confirmed by the present results, that the relaxation mechanisms responsible for the α peaks involve dislocations, it is unlikely that dislocations are taking part in the mechanisms causing the β peaks. If, however, dislocations were involved, two possible mechanisms that could be operating are the pure dislocation relaxation (Bordoni type), or the dislocation-point-defect relaxation (Hasiguti type), discussed in the previous section.

TABLE III Comparison of the behaviour of α and β peaks in cold-worked niobium.

Condition	α peak	β peak
Annealed at 2350° C	None	0.7×10^{-4}
Deformed at 20° C	Height proportional to strain (maximum height 5×10^{-4})	None
Annealed at 70° C	Unchanged	9.0×10^{-4} independent of pre-strain
Annealed at 70 to 240° C	Decreases	Increases
Impurity content	Dependent	No definite relationship

Bordoni mechanism The strength of the dislocation relaxation mechanism is proportional to the dislocation density and the third power of the average loop length [4]. Therefore, the non-existence of the β peak after room-temperature deformation and the independence of the peak height from the degree of strain and impurity content cannot be accounted for by any pure dislocation relaxation process. Furthermore, the increase of the relaxation strength of the β peaks on annealing up to about 100° C, and their subsequent stability up to about 240° C, occur over the temperature range at which migration of oxygen to dislocations is taking place [11]. The migrating oxygen reduces the loop length of dislocations by forming Cottrell atmospheres, which are strong pinning points. Consequently, in terms of the pure dislocation mechanism, the β-peak height should decrease. In view of the above discussion, it could be considered that the α peak is due to the relaxation of pure dislocations and the β peaks are due to dislocation-point-defect relaxations. This situation would be the parallel of the Bordoni and Hasiguti peaks in fcc metals.

Hasiguti mechanism A quick comparison of the Hasiguti peaks and the β peaks shows the following.

(*a*) The Hasiguti peaks increase and saturate or decrease with increasing strain. The β peaks appear to be independent of the strain.

(*b*) The Hasiguti peaks grow and decay at the same temperature, which in all cases is well below the Bordoni-peak annealing-out temperature. The β peaks grow, whilst the α peak is decreasing, and remain stable over a range of about 100° C.

(*c*) The Hasiguti peaks were reduced in height by 0.3% strain at liquid-nitrogen temperature (below the peak temperature). The β peaks were reduced during room-temperature annealing after 0.3% strain at room temperature (above the peak temperature).

These differences are marked enough to lead one not to expect the Hasiguti model to be applicable to the β peaks. For instance, a dislocation-point-defect model cannot account for the independence of the relaxation strength of the β peaks from the amount of cold-work and impurity content. Increasing strains up to 5% should provide more point defects and dislocation loops without detrimental decrease in loop length between strong pinning points, thus giving rise to higher relaxation strengths. Also, for a given strain in a specimen containing fewer interstitial impurities, loops between strong pinning points should be longer than in a specimen of higher impurity content; the former should, therefore, exhibit a higher peak. Furthermore, on annealing within the temperature range from 120 to 240° C, at which oxygen migrates to dislocations forming Cottrell atmospheres [11], the relaxation strength of the β peaks should decrease, as does the α peak (fig. 5).

Point-defect mechanism The possibility of the β peaks being caused by relaxation mechanisms involving only point defects requires the consideration of two types of point-defect configuration: firstly, the stress-induced motion of single interstitials (single vacancies, having cubic symmetry, cannot give rise to relaxation); secondly, the stress-induced motion or rotation of di-vacancies or di-interstitials, as proposed by Dautreppe *et al* for irradiated iron [14], or point-defect/interstitial-impurity-atom pairs, as suggested by Stanley and Szkopiak for irradiated niobium [8]. Since the β peaks do not exist after deformation at room temperature but develop on subsequent annealing at higher temperatures (figs. 4 and 5), it appears that the operating relaxation mechanisms involve point-defect complexes, which are formed at higher temperatures than those at which the point defects were created by deformation. This rules out the single interstitial.

A definite relationship between the height of the β peaks and the degree of strain or the interstitial impurity content, greater than a few atomic parts per million, is not expected in the point-defect model for the β peaks. Increasing the amount of strain and interstitial impurity content should provide more dislocation intersections for point-defect generation. But, at the same time, the increased number of dislocations will provide more sinks for the point defects migrating at the annealing temperature (70° C), thus reducing the number of point-defect complexes that can be formed. The increase in the peak height on further annealing up to about 100° C (fig. 5) is due to continued formation of the point-defect complexes from the remaining free point defects. The decrease of the height of the peaks on annealing at temperatures higher than 240° C could be due to: (i) the break-up of the point-defect complexes; (ii) their agglomeration into non-relaxing clusters; or (iii) their migration to dislocations or other sinks.

Effect of re-straining and re-annealing Further evidence against both the dislocation and dislocation-point-defect hypotheses is provided by the effect on the main β peak of 0.3% straining and 70° C annealing cycles (figs. 6 and 7). On the dislocation hypothesis, the decrease during room-temperature annealing after the 0.3% strain could be attributed to newly generated point defects migrating to the dislocations, thus reducing their loop length; whilst the increase in the peak height after re-annealing at 70° C could be attributed to the annihilation of these point defects at jogs. However, when the re-straining and re-annealing cycle is repeated, approximately the same decrease and increase in peak height should be observed, since the process would be expected to be reversible; but the results in table II show significantly smaller changes in the effect on the second re-straining and re-annealing cycle.

In terms of the dislocation-point-defect hypothesis, an additional 0.3% strain should disturb the relaxation mechanism by removing some of the dislocations from their associated point defects. Annealing at room temperature may allow the point defects to return to the dislocations restoring the relaxations. This would indicate an immediate decrease of peak height, followed by a gradual return to its original height on annealing at room temperature. Again, the process should be reversible, which is not the case.

If, however, a point-defect model is assumed, the decrease after the additional straining can be attributed to the presence of dislocations in the vicinity of the point-defect complexes causing the peak. Because of the proximity of the point-defect complexes to dislocations, the attractive forces between them could cause the point defects to migrate to the dislocations, resulting in the gradual decrease of the peak height at room temperature. The linear dependence of the percentage decrease in the height of the β peaks, with pre-strain before the 70° C annealing, can then be explained on the basis that the specimens pre-strained to higher strains contain higher dislocation densities, and, therefore, a greater number of dislocation lines will be moved during the additional strain. Thus, more point-defect complexes will be close enough to migrate to dislocations at room temperature.

Re-annealing at 70° C allows the point defects, which were generated during re-straining, and some of those point defects which did not reach traps, dislocations or other point defects during the first anneal at 70° C, to form complexes, increasing the number of configurations which contribute to the relaxation peak. Specimens with higher pre-strains, and therefore containing more point defects, have a greater concentration of free point defects remaining after the first anneal and re-strain. (This is because the peak is not fully developed on annealing for 2 h at 70° C (fig. 5).) Thus the peak will increase to higher values in these specimens, since more relaxation configurations can be formed during the second anneal at 70° C. This process is not exactly reversible on the second cycle of re-straining and re-annealing, since the number of free point defects left to form new relaxation complexes has been appreciably reduced by the previous annealing treatments at 70° C.

5. Conclusions

(*a*) Niobium deformed (in tension) at room temperature exhibits a broad internal-friction peak at about 110° K, at a frequency of oscillation of 1 c/sec. This peak has been identified as the α peak previously reported in bcc transition metals.

(*b*) The height of the α peak increases with the degree of deformation and, for a given strain, it decreases with increasing interstitial impurity content.

(*c*) The α peak is attributed to relaxation processes involving dislocations, based either on the Bordoni- or Hasiguti-type mechanisms.

(*d*) On annealing the room-temperature-deformed metal for 2 h at 70° C, a group of peaks occurs at about 200° K. These peaks have been identified as the β peaks observed in bcc transition metals.

(*e*) In the ranges investigated, no definite relationship has been found between the height of the β peak and the degree of deformation or the interstitial impurity content.

(*f*) It has been shown that the β peaks are caused by relaxation mechanisms not involving dislocations, and that the variation of the relaxation strength of the peaks with annealing and re-straining are best accounted for in terms of point-defect complexes. The configuration of these complexes may well consist of vacancies and/or interstitials paired in some way with interstitial impurities.

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