THE \( \nu \) PEAK IN DEFORMED HIGH-PURITY \( \alpha \)-IRON STUDIED BY FORCED VIBRATIONS OUT OF RESONANCE

H. Mizubayashi, H. Kronmüller, A. Seeger

To cite this version:

H. Mizubayashi, H. Kronmüller, A. Seeger. THE \( \nu \) PEAK IN DEFORMED HIGH-PURITY \( \alpha \)-IRON STUDIED BY FORCED VIBRATIONS OUT OF RESONANCE. Journal de Physique Colloques, 1985, 46 (C10), pp.C10-309-C10-312. <10.1051/jphyscol:19851069>. <jpa-00225454>

HAL Id: jpa-00225454
https://hal.archives-ouvertes.fr/jpa-00225454
Submitted on 1 Jan 1985

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
THE \( \gamma \) PEAK IN DEFORMED HIGH-PURITY \( \alpha \)-IRON STUDIED BY FORCED VIBRATIONS OUT OF RESONANCE

H. Mizubayashi\(^*\), H. Kronmüller and A. Seege

Max-Planck-Institut für Metallforschung, Institut für Physik, 7000 Stuttgart 80, F.R.G.

Abstract - The \( \gamma \) peak in high-purity \( \alpha \)-iron was investigated using frequencies over three decades, i.e. 7.4Hz, 0.015Hz and 0.007Hz. For 7.4Hz, the usual resonant vibration mode was used and for 0.015 or 0.007Hz, the forced vibration mode was applied. The observed values of the enthalpy, \( H \), and of the pre-exponential factor, \( 1/\tau_0 \), of the \( \gamma \) peak both showed the tendency of increase with annealing after the low temperature small deformation, i.e. from 0.825eV and \( 4.0 \times 10^{13} \text{s}^{-1} \) to 0.910eV and \( 1.2 \times 10^{15} \text{s}^{-1} \), respectively. From the amplitude dependence, \( 2H_k \) was estimated as about 0.940eV, where \( H_k \) denotes the enthalpy of an isolated single kink in screw-dislocations in \( \alpha \)-iron.

I - INTRODUCTION

In deformed high-purity bcc metals, the so-called \( \alpha (\alpha') \) and the \( \gamma \) peaks are commonly observed and are believed to be associated with intrinsic motions of dislocations (see reviews/1,2/ and also /3/). Among them the \( \gamma \) peak is considered to be the relaxation process of screw dislocations through kink pair formation (KPF)/4/. However, the present knowledge of the activation parameters, the activation enthalpy, \( H \), and the pre-exponential factor, \( 1/\tau_0 \), of the \( \gamma \) peak or of KPF is very limited yet. The aim of the present work is to investigate these activation parameters of the \( \gamma \) peak in deformed high-purity \( \alpha \)-iron.

In deformed high-purity \( \alpha \)-iron, Shimada and Sakamoto/5/ observed the \( \gamma \) peak at about 340K(\( \approx 1 \text{Hz} \)) and obtained \( 0.78 \pm 0.1 \text{eV} \) for \( H \) and \( \log(1/\tau_0)=12.4\pm1.5(1/\tau_0 \text{ in s}^{-1}) \). The peak temperature, \( T_p \), of the \( \gamma \) peak reported in literatures, however, shows a strong dependence on the authors or on the experimental conditions used, e.g. at about 300K(0.5Hz) by Hivert et al./6/ and at about 290K(\( \approx 2 \text{Hz} \)) by Matsui and Schultz/7/. These results suggest that the values of \( H \) and of \( 1/\tau_0 \) themselves may depend on the experimental conditions used as was also suggested by theoretical studies/8,9/.

An investigation covering wider frequency range gives more precise values of \( H \) and of \( 1/\tau_0 \). For the relaxation processes of dislocations, however, a change of frequencies should be made without any handling effects on a specimen. For such an investigation, a method using forced vibrations out of resonance/10,11/ may be suitable, which itself can cover a very wide frequency range, practically \( 1 \sim 10^{-5} \text{Hz} / \text{Hz} \). In the present work, a hybrid measurement using both the resonant vibration at about

\(^*\)Permanent address: Institute of Materials Science, University of Tsukuba, Sakura-mura, Ibaraki 305, Japan
10Hz (R-mode) and the forced vibrations at the order of $10^{-2}$Hz (F-mode) was performed. Using the present method one can investigate a relaxation process over frequencies of 3 decades without any handling effects and also in reasonable measuring times.

II - EXPERIMENTAL PROCEDURES

The specimen used was a polycrystalline wire of α-iron with RRR=3800. The size was 0.9mm×432mm. Before internal friction (IF) measurements, the specimen was deformed by 2.0% in tension at room temperatures (RT). Then, a small deformation by 0.12% in torsion at 80K and warming up to ~420K were performed two times successively in order to obtain a good reproducibility of the γ peak/12/. In the followings, this specimen, the specimen A pre-deformed by 2.0% at RT, will be called as A20.

IF-measurements were performed by an inverted torsion pendulum using R-mode at 7.4Hz and also F-mode at 0.015, 0.007 or 0.006Hz. IF was determined from free decay curves in R-mode and from lag angles between the vibrations of the applied force and of the pendulum in F-mode. The strain amplitude, $\varepsilon_0$, was used as 7x$10^{-6}$ but for an amplitude dependence, 28x$10^{-6}$ was also used. During the IF-measurements, the specimen was subjected to an axial magnetic field of 1500e.

It has been reported/5/ that the γ peak in high-purity α-iron is observed with a peak height, $Q^{-1}_p$, of about 4×$10^{-3}$ immediately after a low temperature small deformation, say the 'virgin' γ peak, but with a much decreased $Q^{-1}_p$ after warming up to ~400K due to recovery and/or pinning phenomena, say the 'annealed' γ peak. In the present work, both the 'virgin' and the 'annealed' γ peaks were investigated.

The measuring conditions for the 'virgin' γ peak were as follows: (1) The specimen, A20, was deformed by 0.05% in tension at 80K (in the followings, 0.05%/80K) and then, (2) a warming up measurement was performed up to about 420K. These procedures were repeatedly performed for various frequencies. For the 'annealed' γ peak, after (2), the repeated measurements between 80K and 390K but without 0.05%/80K) were made for various frequencies. A20 was firstly subjected to the investigation of the 'virgin' γ peak with $\varepsilon_0=7x10^{-6}$. These measuring runs will be called as A20-1 and so on. After A20-1, the specimen was once warmed up to 500K and then, the 'virgin' γ peak was again investigated with $\varepsilon_0=7x10^{-6}$, i.e. A20-2. Following A20-2, the 'annealed' γ peak was investigated with the same $\varepsilon_0$, i.e. A20-2a. The similar investigations as were made in A20-2 and A20-2a were also performed but with the increased $\varepsilon_0$ of 28x$10^{-6}$, i.e. A20-3 and A20-3a, respectively. After A20-3a, $\varepsilon_0$ was decreased to 7x$10^{-6}$ and the investigation of the 'annealed' γ peak was continued, i.e. A20-3b.

III - EXPERIMENTAL RESULTS AND DISCUSSION

Fig.1(a) and (b) show examples of the results observed at 7.4Hz and 0.015Hz for A20-1, respectively. In Fig.1(a), observed changes of period, P, and of IF, $Q^{-1}$, were shown against temperatures, T, by the symbols for the warming rate, $\dot{T}$, of 3K/min or of 10K/min. Here, the 'virgin' γ peak was observed at about 347K with $Q^{-1}_p$ of about 4×$10^{-3}$. For the detailed investigation, the observed γ peak was compared with the calculated one drawn by the solid curves and also with a single Debye peak by dotted and broken curves. For the calculated γ peak, it was assumed that the γ peak can be explained by a broadened Debye peak, where the relaxation strength, $\Delta$, has a Gaussian distribution on logarithms of the pre-exponential factor around 1/$\tau_0$. 1/$\tau_0$ used was the value obtained in Fig.3. The background values used were shown by the broken lines. One can see in Fig.1(a) that the observed γ peak was of a nearly single Debye peak. Further at the higher temperature side, a decrease of the γ peak was started. Such the decrease of the γ peak is commonly observed in high-purity bcc metals and is surmised to be due to a rearrangement of dislocation structures introduced by the low temperature deformation/3/. Here, the observed γ peak seen in Fig.1(a) was overlapped by the early stage of the recovery. For such a case, one can expect that with increasing $\dot{T}$, the temperatures of recovery increases and at the limit of infinite $\dot{T}$, the true 'virgin' γ peak may be obtained. Indeed in Fig.1(a), one can see from the curves for P that the temperature of recovery was increased from ~360K for 3K/min to ~370K for 10K/min, and also from the curves for $Q^{-1}$ that $T_p$ and $Q^{-1}_p$ were increased from 346.1±1K and ~3.9×$10^{-3}$ for 3K/min to 348.3±1K and ~4.4×$10^{-3}$ for 10K/min, respectively. From the repeated measurements with $\dot{T}$ in between 3K/min and 12K/min, the true $T_p$ and $Q^{-1}_p$ for 7.4Hz in A20-1 were determined as 349.0±1K and ~4.7×$10^{-3}$, respectively. The 'virgin' γ peak for 0.015Hz seen in Fig.1(b) was observed at 284.8±1K with $Q^{-1}_p$ of...
The peak profile was almost the same as that seen in Fig.1(a). For the 'virgin' γ peak for 0.015 or 0.007Hz, small dislocation pinning were observed at the lower temperature side. Therefore, the measurements were performed with a very low T to complete the dislocation pinning far below Tp. Besides the γ peak, one can also see a small shoulder at about 230K. From the repeated measurements, Tp's determined were 284.6±1K for 0.015Hz and 277.4±1.5K for 0.007Hz, respectively, and Q-lp averaged over for 0.015Hz and 0.007Hz was \( \sim 5.2 \times 10^{-3} \) in A20-1.

Similar investigation as mentioned above was also made in A20-2 and in A20-3. The obtained results for Tp vs frequency, f, for the 'virgin' γ peak are seen in Fig.3. Fig.2 shows examples of the 'annealed' γ peak observed at 7.4Hz and at 0.015Hz in A20-2a after A20-2. For the repeated measurements following 0.05%(80K), the 'annealed' γ peak showed transient changes of Tp and Q-lp in early runs, therefore these values were determined from the results obtained after the transient changes were completed. Similar investigation was also performed in A20-3a and in A20-3b after A20-3, and the obtained results for Tp vs f for the 'annealed' γ peak are also shown in Fig.3.

For a relaxation peak, the relation of Q-lp = 1/Tp can be expected/10/. In the present work, the observed ratio of (Q-lp for 0.015 and 0.007Hz / Q-lp for 7.4Hz), Q-lp-ratio, was always found in between 1.0 and 1.3 except 1.8 in A20-3 and that of (Tp for 7.4Hz / Tp for 0.015 and 0.007Hz), Tp-ratio, was always about 1.2. That is, the relation of Q-lp-ratio = Tp-ratio was observed within experimental errors through the present work, but in A20-3, Q-lp for 7.4Hz was much smaller than that expected from the results for 0.015 and 0.007Hz. Combining these results with the facts that the 'virgin' γ peak for 7.4Hz was partly overlapped by the recovery process and in A20-3, the increased c0 of 28x10^-6 was used, one can say that the increased c0 might accelerate the recovery process. Therefore, H and 1/\( \tau_0 \) in A20-3 obtained in Fig.3 will not be discussed and on the other hand, one can say that the recovery was more proceeded after A20-3, i.e. in A20-3a and A20-3b, than after A20-2, i.e. in A20-2a.

Fig.4 shows the plot for log(1/\( \tau_0 \)) vs H which were obtained in Fig.3. From the amplitude dependence investigated in A20-3a and A20-3b, increases of both H and 1/\( \tau_0 \) with decreasing c0 can be seen. Combining this amplitude dependence of H with Seeger's theory on KFP-process/8/, 2Hk was estimated as about 0.940eV, where Hk denotes the enthalpy of an isolated single kink in screw-dislocations in α-iron. On the other hand, for the effect of annealing which was investigated in A20-1 or A20-2, A20-2a and A20-3b, one can see the tendency of increases of both H and 1/\( \tau_0 \) with annealing proceeded. Further, comparing the effect of annealing seen between in A20-2a and in A20-3b with that of amplitude, in A20-3a and in A20-3b, one can see that these effects were almost the same for A20. This result can be reasonably understood after the following consideration; the internal stress applied to the 'virgin' γ peak here might be mainly introduced by 0.05%(80K) and be decreased with annealing. Therefore, it is concluded in the present work that both H and 1/\( \tau_0 \) increase with decreasing stress applied to the γ peak process. Detailed results including the effect of pre-deformations at RT and further discussions will be reported elsewhere.

ACKNOWLEDGEMENTS

The authors wish to express their gratitude to Messors H. Waldmann and J. Wolf for their invaluable helps throughout the experiments and to Professors H. Schultz and S. Okuda for discussions. One of the authors (H.M.) acknowledges deeply financial support from the Alexander von Humboldt Foundation.

REFERENCES

Fig.1 Examples of the 'virgin' γ peak in A20-1 for 7.4 Hz (a) and for 0.015 Hz (b) with $\varepsilon_0=7\times10^{-6}$ (see text).

Fig.2 Examples of the 'annealed' γ peak in A20-2a for 7.4 Hz and for 0.015 Hz with $\varepsilon_0=7\times10^{-6}$ (see text).

Fig.3 Arrhenius plot for the γ peak observed for various experimental conditions.

Fig.4 Log(1/\tau_0) vs. H, which were obtained in Fig.3.