An Internal Friction Study of Hydrogen in Cyclotron Irradiated Va Transition Metals

著者	Hanada R.
journal or publication title	CYRIC annual report
volume	1982
page range	43-45
year	1982
URL	http://hdl.handle.net/10097/48690

CYRIC
Annual Report 1982

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I. Introduction

For the development of the constituent materials for the future fusion reactor, which will be attacked by both hydrogen isotopes and the irradiation damage, the basic knowledge on the nature of the interaction between lattice defects and hydrogen isotopes will become necessary. To obtain a clue for such an investigation, an internal friction (I.F.) measurement was performed on cyclotron irradiated Va transition metals doped by either hydrogen (H) or deuterium (D). Also for the purpose of comparison, measurements on cold-worked specimens were performed, of which results will be described in the followings.

II. Experimental

(i) Specimen

A high purity niobium (Nb) specimen was prepared by annealing in an ultra high vacuum (10^{-8} Torr.) at a temperature close to the melting point. Subsequently it was doped by nitrogen (N) to the concentration of 0.5 at %. The aim of the N doping is to reduce the effect of the residual H, which is inevitably present in an UHV annealed Nb and causes a large hydrogen cold work peak (H.C.W.P.). Next, specimens were electrolytically doped either by H or D.

(ii) Irradiation

Several ^{4}He irradiations were performed by the A.V.F. cyclotron with the energy of 50 MeV with the current density of 0.2-0.5 $\mu A/cm^2$. The total dose was between 10^{16} and $4\times10^{16}/cm^2$. Specimens were glued to the one side of a grooved copper plate, of which the other side was cooled by liquid nitrogen. The direct measurement of the specimens temperature during the irradiation was difficult, though a thermo-couple soldered to the cooper plate showed the temperature increase during the irradiation was kept less than 30 k.

(iii) I.F. Measurement

After the irradiation, specimens were warmed up to the room temperature and the I.F. measurement was performed by a conventional torsion pendulum apparatus with the measuring frequency of 0.7 Hz. Measurements were performed between 20 and 270 K. It took about 1 day to complete one running.

III. Results

Figure 1 shows the result for a Nb-N-D specimen irradiated to a dose of $10^{16}/cm^2$. A clear peak at 75 K is the N-D pair peak, which is caused by the D jump around N impurity atom and not related to the irradiation induced defects. However, a small hump is present between 100 and 120 K only in the irradiated specimen. To confirm the hump is related to the irradiation induced defects, the same measurement was performed on a Nb-N-H specimen irradiated to $4\times10^{16}/cm^2$, which is about 4 times higher dose than that for Fig. 1. Figure 2 shows the result for the Nb-N-H, where a clear peak develops at about 100 K. Although not shown in Fig. 2, the peak is found to take place only after the H doping to the irradiated specimen. The growth of the peak with the increasing dose and the absence of it without H doping suggest strongly that it is caused by a kind of interaction between H and the radiation induced defects. The hump at 55 K is due to the N-H pair. The large one at about 230 K, which is present only in the run (b) and hence not reproducible and also present sometimes in non-irradiated specimens, is not related to the irradiation induced defects. The curve (c) in Fig. 2 shows the results after the cold work at R.T. to the S.S.S. of 30 %. The peak at 100 K is the H.C.W.P., which has been confirmed to be caused by an interaction between H and dislocation introduced by the cold work.

IV. Discussion

Recent I.F. measurements on high purity Va transition metals have revealed that a very small amount of H (less than a few 10 at ppm) is sufficient to cause a large cold work peak, which is now renamed as H.C.W.P. instead of the name of α peak. However recent experiments by the present author have shown that the N or O is effective to suppress the H.C.W.P. and a severe cold work is necessary to cause the H.C.W.P. as shown in Fig. 2. For the case of a high purity specimen, which usually contains H, a very small cold work, which is inevitable in the specimen setting, is sufficient to cause a noticeable H.C.W.P. Therfore with using a high purity specimen, it is difficult to distinguish between the radiation effect and the handling effect. However for the case of the N doped specimen handling cold work is not sufficient to cause the H.C.W.P. Therefore, the present author considers the 100 K peak in the irradiated specimen as caused by an interaction between radiation induced defects and H.

The close similarity of the H-defects peak and the H.C.W.P. suggests that the defects cluster, which is formed after the R.T. annealing, interacts with H as if dislocations do.

A similar I.F. peak has been found in neutron irradiated Nb,1) although the 100 K peak has been interpreted as dislocation-radiation induced point defects interaction peak. The present work suggests the 100 K peak should be re-interpreted as the defects clusters-H interaction peak.

V. Acknowledgement

The author would like to thank to the staff of the Cyclotron and Radioisotope Center for their operation of the cyclotron and for their skilful beam transport. Also the author would like to thank to Dr. S. Takaki (RIISOM) for his helps during the irradiation.

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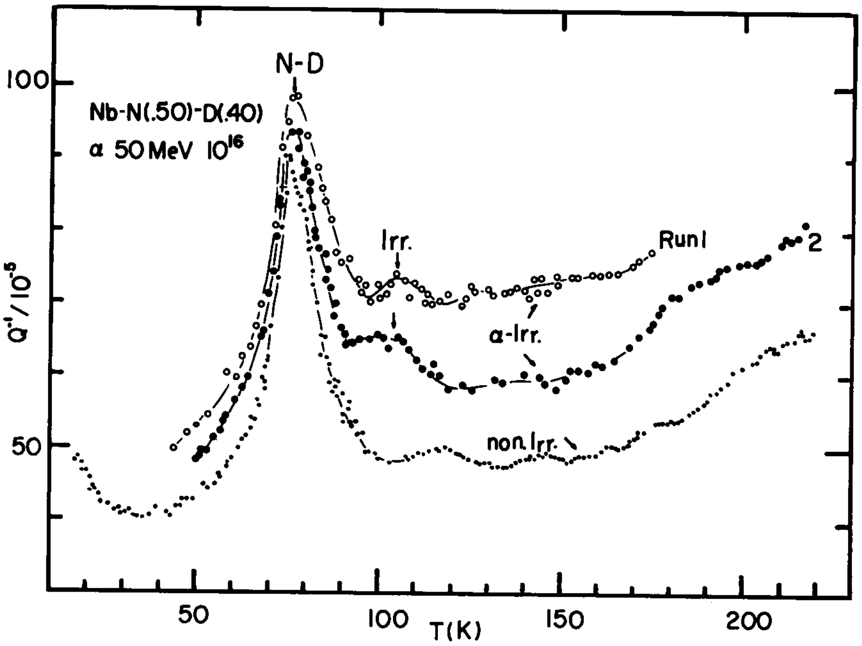

Fig. 1. Results for I.F. measurements on α-irradiated Nb doped by N and D. (Run 1 and Run 2) A result for a non-irradiated specimen is also shown (Bottom).

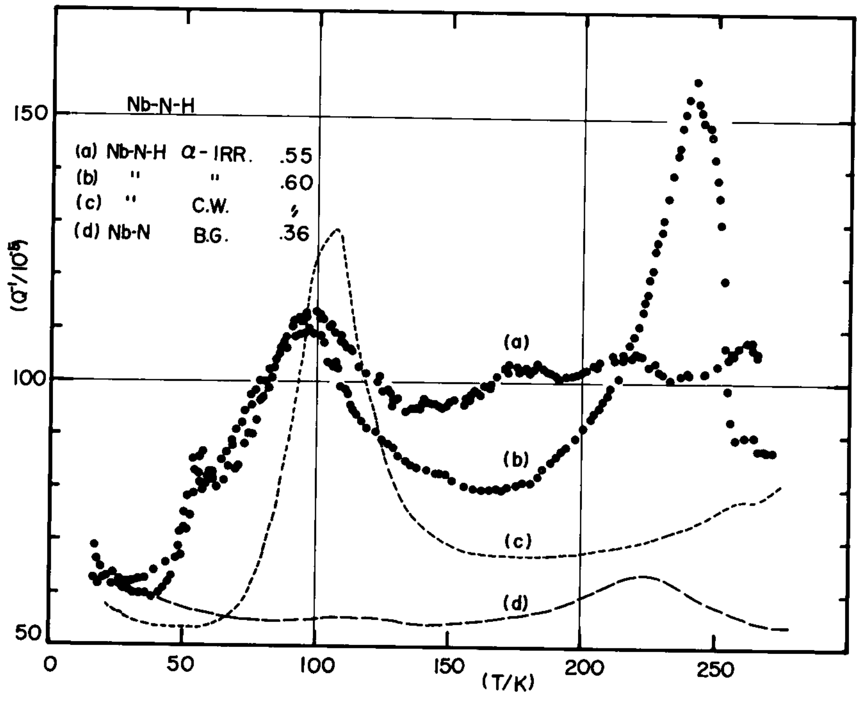

Fig. 2. Results for Nb-N-H ((a) and (b)). For comparison the result after the only cold work (c) and in the annealed state (d) are shown.