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Helium-vacancy clustering in V– 4Cr– 4Ti at elevated temperatures

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Abstract

Thermal Helium Desorption Spectrometry was used to investigate the helium-vacancy clustering in V–4Cr–4Ti at various implantation temperatures. Samples were implanted with 1 keV helium to a dose of 10^{14} cm^{-2} at implantation temperatures ranging from 300 to 700 K. Helium trapping inside the sample was almost completely suppressed at implantation temperatures above 700 K. Helium-vacancy clustering at gas impurities was not observed at temperatures above 500 K. This result is consistent with the earlier proposed model where the helium-vacancy-oxygen complex is unstable above 550 K. Helium trapping at 500–700 K is ascribed to pre-existing traps, e.g. fine-size precipitates. In a number of experiments the implantation at elevated temperature was preceded by room temperature (RT) pre-implantation with doses varying from 1.8×10^{13} to 10^{14} cm^{-2}. It was observed that the clusters created during the RT implantation were stable enough to provide further cluster growth during the subsequent high temperature (700 K) implantation.

1. Introduction

Vanadium based alloys are now considered as candidate structural materials for fusion applications [1–4]. Therefore, during the last years, a considerable effort is put into the investigation of defect formation under implantation. According to the review of Matsui et al. [1] V–4Cr–4Ti is now selected as a reference material for further investigation. In the Dynamical Helium Charging Experiments, Chung et al. [3] have shown that neutron irradiation up to 31 dpa at 500–600°C in the presence of helium does not result in the formation of helium bubbles. Possible explanation for this fact was presented in the thermal helium desorption work [4] where the nucleation sites for helium-vacancy clustering were observed not to be stable at 550 K. The present work aims to investigate the process of nucleation of helium-vacancy clusters at elevated temperatures.

2. Experimental

The THDS experiments were carried out in a UHV chamber equipped with an ion gun with an ExB filter and a calibrated mass-spectrometer. Vacuum inside the chamber during the measurements was 10^{-8}–10^{-7} Pa. First, the sample under investigation was implanted with 1 keV helium and then annealed with a constant annealing rate of 10 K/s. During the annealing helium release was monitored by the mass-spectrometer. A detailed description of the THDS technique is presented by van Veen in [5]. According to TRIM calculations, [6] 1 keV helium implantation with a typical implantation dose of 10^{14} cm^{-2} corresponds to 0.01 dpa level with 1500 ppm helium located at a depth of about 9.3 nm.

The V–4Cr–4Ti samples were obtained from Chung (ANL) with the heat reference number BL-71. Before the measurements, the samples were electrochemically polished in sulphuric acid and then annealed in a vacuum of 10^{-7} Pa at 1600 K for 10–20 min.

3. Results and discussion

Typical thermal desorption spectra after 1 keV helium implantation at room temperature (RT) are shown

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in Fig. 1. The implantation dose is varied from 3.2×10^{13} to 3.2×10^{14} cm^{-2}. During the implantation phase helium is trapped at pre-existing traps or at the defects created by the implantation. During thermal annealing, the helium traps dissociate according to their dissociation temperature contributing to the helium release. In the case of first-order release, the dissociation energy of the helium trap is directly related to the desorption temperature by the following equation.

$$\frac{E^{\mathrm{diss}}}{kT^{2\mathrm{max}}} = \frac{v}{\beta}\exp\left(-\frac{E^{\mathrm{diss}}}{kT_{\mathrm{max}}}\right),$$

where E^{diss} is the dissociation energy, T_{max} is the temperature of the release peak, v is the attempt frequency, usually, taken as a Debye frequency $v = 10^{13}$ s^{-1}, $\beta = 10$ K/s is the annealing rate and k is the Boltzmann constant. Fig. 1 demonstrates that the thermal stability of the defect clusters contributing to the release spectra increases with the implantation dose. In the earlier work, [4] a certain relation between the release peaks and the structures of the defect clusters was established. The model was supported by the computer simulations carried out with the Monte-Carlo program MODEX [7]. According to this model, the first release peak at 550 K corresponds to the He_n–V–X-type defect cluster, where He is a helium atom with $n > 1$-the number of helium atoms, V denotes vacancy and X = C, N, O denotes oxygen, nitrogen or carbon impurity atom. The defects are formed during the implantation according to the following chain of reactions:

$$V^* + X \rightarrow VX,$$

$$He^* + VX \rightarrow HeVX,$$

$$He^* + HeVX \rightarrow He_2VX,$$

where the asterisk denotes mobility at the implantation temperature. With the increase of the implantation dose, the $He_nV_mX_k$-clusters become bigger, and according to the THDS spectra in Fig. 1, they appear to be more stable when the cluster size increases. However, the cluster build-up process can proceed only under the condition that the VX and HeVX-nucleation centres are already created. Keeping in mind that these centres are not stable at 550 K [4], the cluster formation process at elevated temperatures should be suppressed.

The THDS spectra obtained after 1 keV helium implantation to a dose of 10^{14} cm^{-2} at various

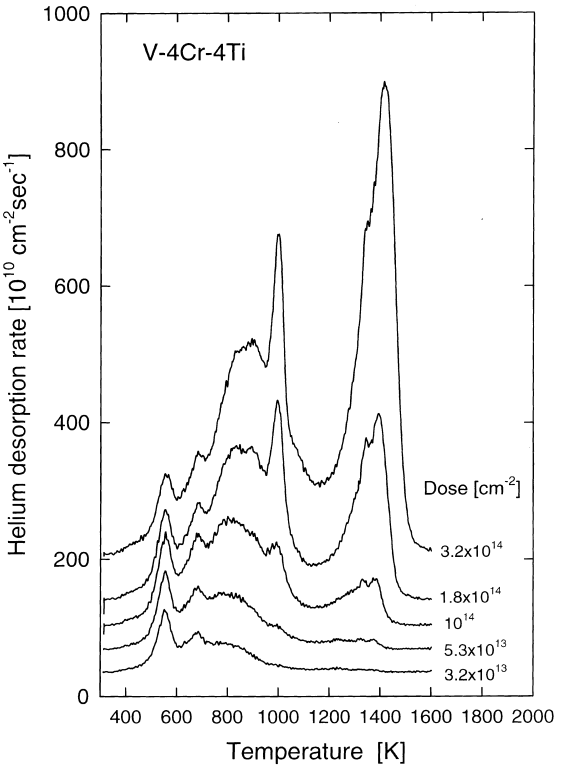

Fig. 1. Thermal helium desorption spectra for V–4Cr–4Ti after 1 keV helium implantation obtained for the implantation doses ranging from 8×10^{12} to 3.2×10^{14} cm^{-2}.

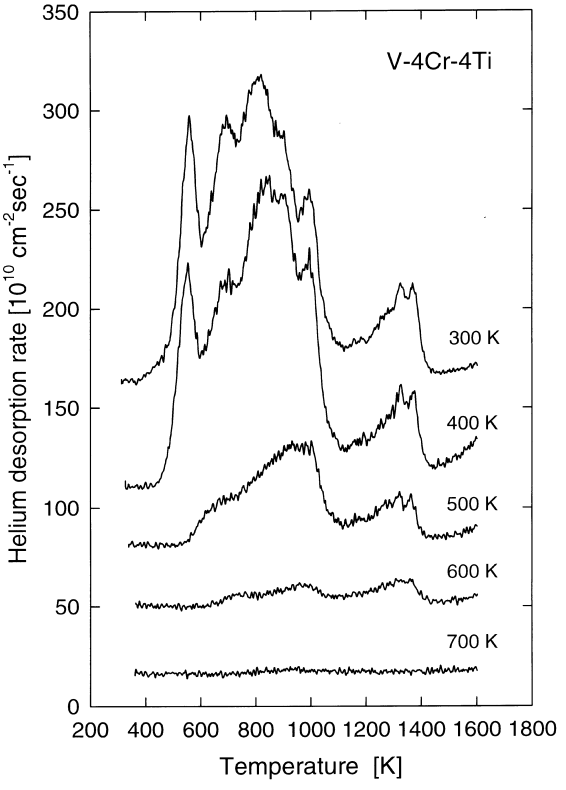

Fig. 2. Thermal helium desorption spectra for V–4Cr–4Ti after 1 keV helium implantation with the implantation dose 10^{14} cm^{-2}. The implantation temperature varied from 300 to 700 K.

temperatures are shown in Fig. 2. Note that already during the implantation at 700 K no defect clusters are created, including those that are unstable at this temperature, as well as the clusters which dissociate at higher temperatures.

In another set of experiments, the 700 K implantation was preceded by RT pre-implantation to various doses. The implantation energy in both cases was 1 keV. The results are shown in Fig. 3. The spectra (a)–(c) show helium release after the RT implantation to the indicated doses and the subsequent ramp annealing to 700 K. Therefore, the observed helium release in the spectra (a)–(c) (indicated as the *survival area*) is ascribed to the clusters which are stable at 700 K. The spectrum (d) is obtained after implantation at 700 K without RT pre-implantation. No cluster build-up is observed in this case. The spectra (e)–(g) show the helium release when the 700 K implantation was preceded by the RT pre-implantation to the doses of 1.8×10^{13}, 5.3×10^{13} and 10^{14} cm^{-2}, respectively. Since during the subsequent 700 K implantation no new nucleation centres can be created, the only traps for implanted helium must be the clusters from the survival area, shown in Fig. 3(a)–(c). Therefore, care should be taken while controlling the implantation temperature. During a short accidental drop of the temperature below 700 K, nucleation centres can be created capable to provide further clustering at high temperature implantation.

4. Conclusions

1. Nucleation centres for cluster build-up in V–4Cr–4Ti are shown to be unstable above 700 K.

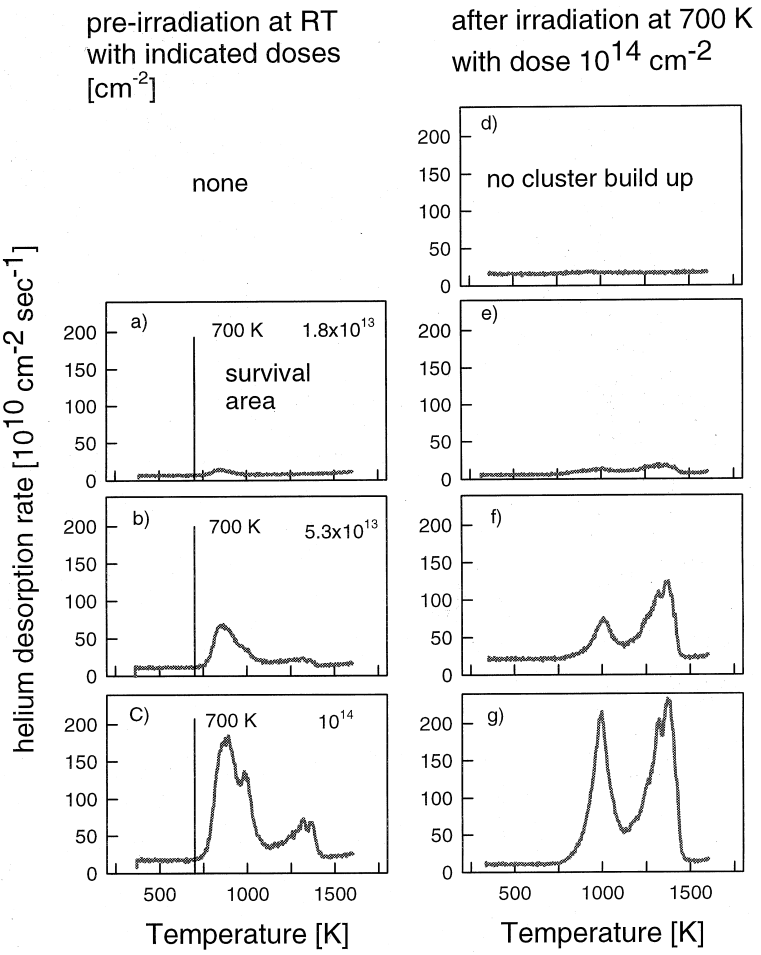

Fig. 3. Thermal helium desorption spectra measured after 700 K 1 keV helium implantation to a dose of 10^{14} cm^{-2} (spectrum (d)). Spectra (e)–(g) show the release when the 700 K implantation was preceded by RT implantation to doses of 1.8×10^{13}, 5.3×10^{13} and 10^{14} cm^{-2}, respectively. The spectra (a)–(c) show helium release from the clusters from the survival area, i.e. the clusters stable above 700 K.

2. The suppression of the cluster build-up at elevated temperatures ($T = 700$ K) was not observed if the nucleation centres stable above 700 K were created by the pre-implantation at RT. The latter observation is important with regard to the temperature cyclic operation of the fusion reactor.

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