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Influence of thermal treatment on helium trapping at fine-size precipitates in V–4Cr–4Ti

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Abstract

Thermal Helium Desorption Spectrometry (THDS) studies carried out on V–4Cr–4Ti alloy have revealed two types of sinks for helium: (1) helium–vacancy clusters nucleated at interstitially dissolved gas impurities, and (2) pre-existing traps, e.g. fine-size precipitates. The effect of thermal treatment on the precipitates was studied by pre-annealing of the samples before the THDS measurements. The pre-annealing was carried out in high vacuum $<10^{-7}$ Pa for 1 h in the temperature interval from 600 to 1700 K. It was observed that helium trapping into the pre-existing traps was dramatically suppressed for annealing at 1200–1300 K. However, a short annealing at 1400 K completely restored the efficiency of the pre-existing traps. Thermally induced modifications of the precipitate–host interface are held responsible for the observed effect.

1. Introduction

Recent studies on vanadium based alloys have demonstrated their excellent behaviour under irradiation [1–3]. Particularly, V–4Cr–4Ti is now considered as a reference material for further investigation. However, it was reported by Matsui [4] that the final thermal treatment had a considerable effect on the ductility. It was shown that V–4Cr–4Ti and V–4Cr–4Ti-0.1Si have a DBTT around 290 K if annealed at 1373 K, while they demonstrated remarkable ductility at 77 K after annealing at 1223 K. Chung et al. [5] have also reported that among the V–(4–5)Cr–(4–5)Ti alloys annealed for 1 h at 1223, 1273, 1323 and 1373 K, the ones annealed at 1273 K showed the optimal impact properties. This observation was valid even for V–5Cr–5Ti which has been reported earlier as having poor mechanical properties. Annealing lower than 1273 K, e.g. 1023 K, was also reported to worsen the material properties [6].

The present thermal desorption study is aimed at investigating the effect of the thermal treatment on helium trapping in the V–4Cr–4Ti alloy.

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. The 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 [7]. According to TRIM calculations [8] 1 keV helium implantation with a typical implantation dose of 10^{14} cm^{-2} corresponds to a 0.01 dpa level with 1500 ppm of 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 of BL-71. Before the measurements the samples were electrochemically polished in sulphuric acid and then annealed in vacuum of 10^{-7} Pa at 1600 K for 10–20 min.

3. Results and discussion

A previous thermal desorption study on V–4Cr–4Ti [1] has demonstrated two different types of traps for

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helium. One contribution to the release spectra was due to dissociation of $He_nV_mX_k$-type defects, where He denotes a helium atom, V denotes a host vacancy, and X = C, N, O are carbon, nitrogen and oxygen impurities. The actual numbers of He's, V's and X's in the cluster n, m and k can vary but are always 1 or more than 1. The helium release from these types of defects is characterised by a number of peaks with the first peak to appear

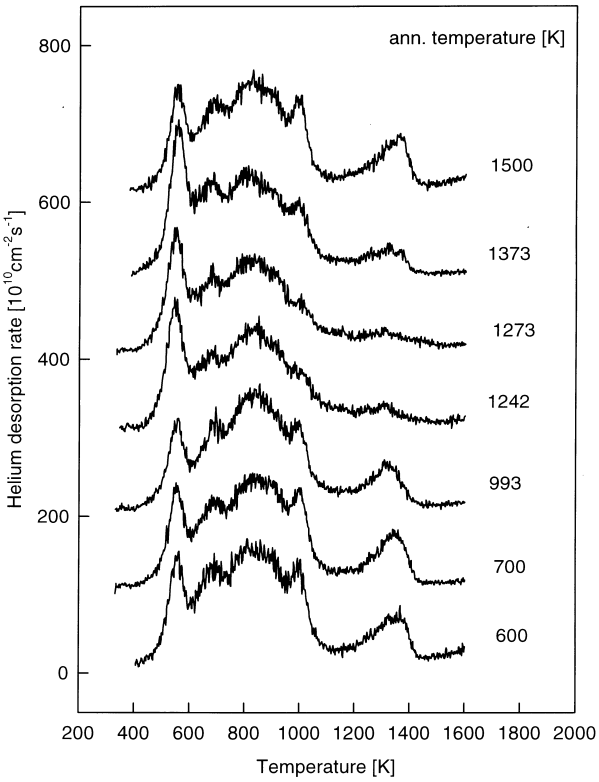

Fig. 1. Thermal desorption spectra for V–4Cr–4Ti after 1 keV helium implantation at a dose of 10^{14} cm^{-2}. Before the implantation the sample was annealed in high vacuum for 1 h at the indicated temperature.

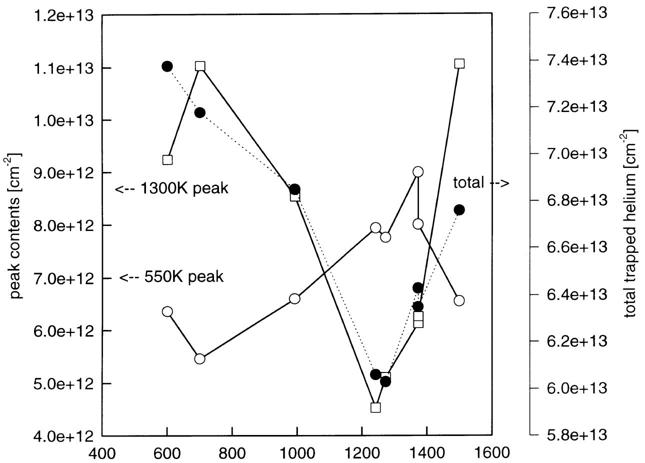

Fig. 2. Contributions of helium release from different temperature intervals versus the pre-annealing temperature.

at 550 K which is ascribed to He_nVX clusters with $n \geqslant 1$. Another contribution to the spectra located in the higher temperature interval from 1200 to 1400 K observed for V–Cr–Ti alloys but not for pure vanadium [1], was ascribed to the release from small helium bubbles formed at the pre-existing traps (PT), e.g. fine size precipitates. Recently it was discovered that this contribution depends on the thermal treatment of the sample before the measurements.

The effect is demonstrated in Fig. 1, where the desorption spectra for 1 keV 10^{14} cm^{-2} helium implantation in V–4Cr–4Ti are presented. Before every implantation the sample was pre-annealed for 1 h at the indicated temperature. The pre-annealing at 1242 and 1273 K dramatically suppresses the helium release at 1000 and 1300 K. The annealings at higher than 1300 K and lower than 1200 K seem to restore the peaks' contributions. The contributions from different temperature regions are plotted in Fig. 2 versus the pre-annealing temperature. It is also seen that a decrease of the 1300 K peak contribution is accompanied by an increase of the 550 K peak related to He–V–X clusters.

The following experiment was done to find out, whether the restoration of the 1300 K peak at low pre-annealing temperatures was due to the pre-annealing or to the fact that during the previous helium desorption measurement the sample was shortly ramp annealed up to 1600 K. The experiment was carried out in the following steps:

(1) The sample was annealed for 1 h at 1252 K to suppress the pre-existing traps responsible for the 1300 K release.

(2) The sample was implanted with 1 keV helium but the subsequent ramp annealing during the measurements was done only till 1370 K (see Fig. 3, spectrum A). As expected the 1300 K peak was suppressed.

(3) The sample was re-implanted with 1 keV helium with the same dose but this time the ramp annealing was done in a common way till 1600 K. The 1300 K peak in the resulting spectrum B is suppressed as well. Note that this time during the desorption a temperature of 1600 K was reached.

(4) The 1 keV helium implantation was repeated and the ramp annealing has demonstrated the restoration of the 1300 K peak (see Fig. 3, spectrum C).

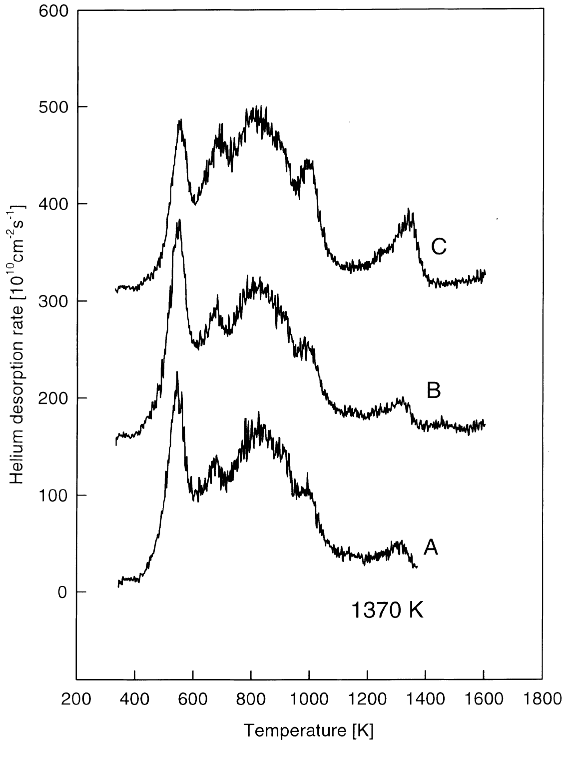

Fig. 3. Thermal desorption spectra in V–4Cr–4Ti after 1 keV helium implantation with a dose of 10^{14} cm^{-2}. Spectra A–C are related to different treatments of the sample described in the text.

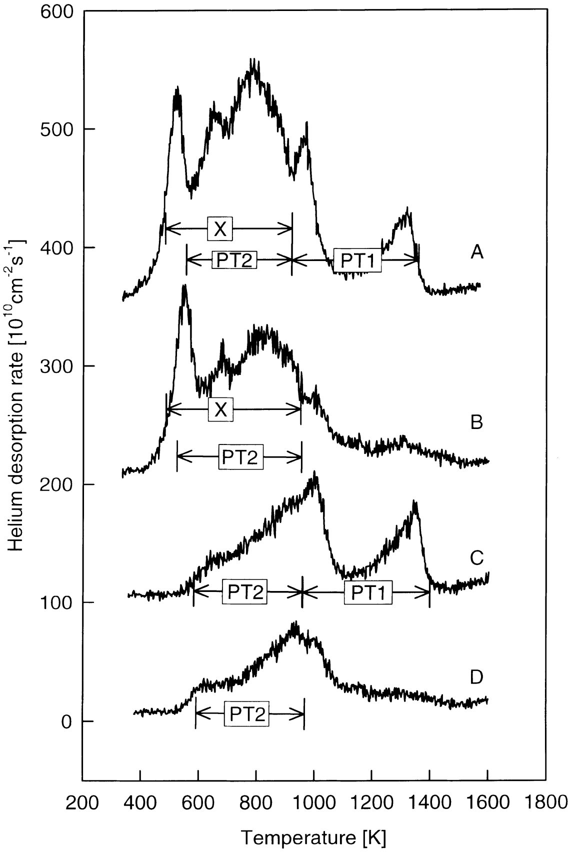

Fig. 4. Thermal desorption spectra in V–4Cr–4Ti after 1 keV helium implantation with a dose of 10^{14} cm^{-2}. The implantation temperature and the thermal treatment of the sample for the spectra A–D are explained in Table 1.

Table 1
The relation between the sample treatment and the corresponding spectra in Fig. 4.

Spectrum	Pre-annealing	Implant temperature	Helium traps
A	>1400 K, 10 s	300 K	X = C, N, O, PT-1, PT-2
B	1273 K, 1 h	300 K	X = C, N, O, PT-2
C	>1400 K, 10 s	500 K	PT-1, PT-2
D	1273 K, 1 h	500 K	PT-2

The helium traps responsible for helium release in the spectra are also given.

Evidently the reason for the restoration of the 1300 K peak in the spectrum C was the annealing of the sample above 1400 K during the previous measurement of the spectrum B.

Fig. 4 summarises the effect of the thermal treatment and the implantation temperature on the desorption spectra. The relation between the sample treatment and the corresponding spectrum is presented in Table 1. The traps that are responsible for the helium release are also shown. Note that in Table 1 two types of pre-existing traps are considered: PT-1 are the traps affected by the 1200–1300 K thermal treatment and PT-2 are not. Four spectra A–D in Fig. 4 are measured after the same 1 keV 10^{14} cm^{-2} helium implantation. The implantations A and B were carried out at room temperature and the implantations C and D were done at 500 K. In the latter case no $He_nV_mX_k$ clusters formation can occur since the VX and HeVX-nucleation centres are not stable [1]. The measurements B and D were done after 1273 pre-annealing for 1 h. During this pre-annealing the pre-existing traps PT-1 which are responsible for the helium release at 1000 and 1300 K disappear. Before the measurements A and C no special pre-annealing was done, but the final thermal treatment in this case was the previous THDS measurement and during this measurement the sample was ramp annealed above 1400 K for about 25 s.

The probable candidates for the pre-existing traps PT-1 and PT-2 can be the fine-size Ti(O, N, C) precipitates reported by Chung et al. [3] or any of the alloying elements: Cr or Ti. Further investigations combined with TEM study are necessary to define the real nature of the pre-existing traps.

4. Conclusions

1. It was shown that a 1 h annealing at 1200–1300 K suppresses helium trapping to the pre-existing traps associated with precipitates in V–4Cr–4Ti alloy.
2. Short annealing at 1400 K restores the pre-existing traps responsible for helium release at 1000 and 1300 K.

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