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Effect of Ti solute on the recovery of cold-rolled V–Ti alloys

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Abstract

The recovery characteristics of cold-rolled pure V and V–Ti alloys with compositions of 0.3, 1 and 4.5 at.% Ti have been investigated by positron annihilation spectroscopy. The recovery is accomplished in two stages. Fifty percent cold rolling induces the formation of microvoids in V–0.3Ti and V–1Ti but not in V–4.5Ti. The first recovery stage in pure V, V–0.3Ti and V–1Ti starts with the dissolution of microvoids. The recovery curves of the annihilation parameters for the alloys indicate the formation of Ti-rich precipitates during the first recovery stage. These precipitates act as very efficient vacancy sinks. The second recovery stage starting for annealing temperatures above ≃1150 K is attributed to annealing of vacancies associated to the precipitates. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The structural materials for fusion technology applications must maintain a good mechanical behaviour under severe conditions imposed on the environment of a fusion reactor. This means to have a high radiation damage resistance along with adequate tensile and creep strengths and ductility at high temperatures. Moreover, these materials must have very low induced activation and afterheat decay to satisfy the requirements of passive safety, waste disposal, maintenance and recycling. Vanadium-based alloys appear to be promising materials due to their high temperature capabilities combined with a good swelling resistance and a low activation [1–4].

In particular, some neutron-irradiated vanadium alloys exhibit very low swelling and reasonable good tensile and fracture properties. Among the binary vanadium alloys, the V–5wt% Ti composition is interesting because of its low ductile–brittle transition temperature (DBTT), very low void swelling and high creep resistance [2–8]. The origin of these useful properties is attributed to the microstructure of defects induced by the Ti atoms. It appears that Ti segregation and fine precipitate formation along dislocations change the vacancy sink strength of dislocations and voids inhibiting the nucleation and growth of voids in vanadium [9–12]. Moreover, the DBTT of vanadium alloys appears to be largely influenced by annealing. There exist results indicating that the DBTT of V–Ti alloys increases after annealing above 1273 K [13]. The origin of the annealing effect on the DBTT has not been investigated although it has been associated to the dissolution of fine size precipitates. The design and development of vanadium alloys for fusion reactors requires a good understanding of the interaction of an oversized solute atom, such as Ti, with vacancies, dislocations, and interstitial impurities.

Positron annihilation spectroscopy (PAS), as a sensitive technique to vacancy-type defects in metals, was recently used to investigate the interaction of Ti solute with vacancies in electron-irradiated V–Ti alloys [14]. The results reveal that Ti solute in vanadium is a very effective trap for vacancies giving rise to different types of vacancy–Ti complexes. Also, PAS has been applied to investigate the recovery of pure vanadium single-crystals deformed under different conditions [15]. In addition, PAS is a technique with capabilities to reveal the presence of NaCl-type metal carbides and nitrides precipi-

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tated in a matrix [16,17]. Positron lifetime experiments have revealed TiC precipitation during the recovery of a cold-worked Ti-stabilized austenitic steel [18,19].

To get insight on the interaction of Ti solute with vacancies and dislocations in vanadium we have investigated the effect of the Ti addition on the recovery of cold-rolled samples using PAS.

2. Experimental

The materials used were 99.9% pure V and V–Ti alloys with Ti concentrations of 0.3, 1.0 and 4.5 at.%. V–Ti alloy buttons were produced by repeated arc melting in a high-purity argon atmosphere. The homogeneity and composition of the alloys were checked by X-ray fluorescence analysis. Slices of the alloys were cold rolled into sheets and solution annealed at 1573 K for 2 h in an oil-free vacuum of $\lesssim 10^{-3}$ Pa. The pure V samples underwent the same heat treatment. After these treatments the mean content of C and O in the samples were, respectively, 340 and 290 wt ppm for the alloys, and 200 and 50 wt ppm for pure V; the N content was always below 20 wt ppm for all the samples. Pairs of samples were gradually cold rolled up to a 50% thickness reduction. After rolling the samples were polished and isochronally annealed for 20 min in steps of 20 K. A pair of undeformed (solution annealed) V–1Ti samples was also heat treated following the above annealing program. The anneals above 420 K were made in an oil-free vacuum of $\lesssim 10^{-3}$ Pa and at temperatures below 420 K in a silicon oil bath.

Positron annihilation experiments were made using a ^{22}Na positron source ($\simeq 8 \times 10^5$ Bq) inside sealed Kapton foils, sandwiched between a pair of samples. Experiments were performed on undeformed and as-rolled samples of the alloys. After each annealing step, positron lifetime measurements at room temperature were performed on the samples. Doppler-broadening measurements of the annihilation peak over the range 10–300 K were done after selected annealing steps. A spectrometer with a time resolution of 230 ps (FWHM) was used for lifetime measurements. The lifetime spectra were analyzed with the programs RESOLUTION and POSITRONFIT in terms of one or two exponential components to obtain the best fit after subtracting background and the source contribution. The two-component spectra were characterized by the mean positron lifetime given by

$$\bar{\tau} = I_1\tau_1 + I_2\tau_2 \tag{1}$$

being I_i and τ_i the intensity and the lifetime of the spectral components, respectively.

Doppler-broadening measurements of the annihilation peak at 511 keV were made with a stabilized HP Ge detector having an energy resolution of 1.62 keV at the 1.33 MeV line of ^{60}Co. The Doppler-broadening was characterized by the lineshape parameter S, defined as the fraction of counts within an energy window of 1.50 keV centered at 511 keV.

3. Results

3.1. Pure V

Fig. 1 shows the effect of the isochronal anneals on 50% cold-rolled V samples. After rolling the positron lifetime spectrum of the samples is single-component becoming two-component for anneals between 423 and 783 K. The lifetime of the second component τ_2 stays constant at (250 ± 20) ps for anneals below 550 K; this lifetime value is characteristic of three-dimensional

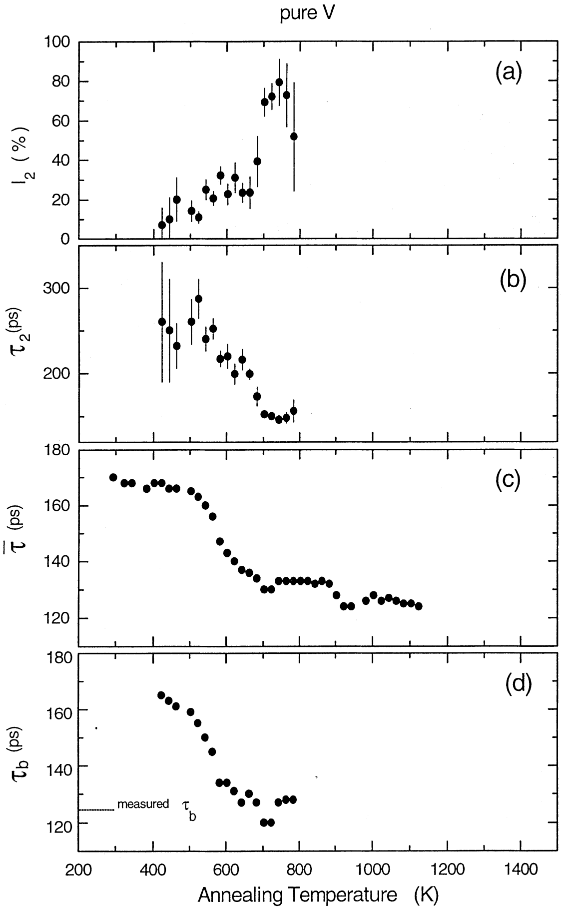

Fig. 1. Positron annihilation parameters versus annealing temperature for 50% cold-rolled pure V. I_2 and τ_2 are the intensity and the lifetime of the second spectral component, respectively, $\bar{\tau}$ is the mean positron lifetime and τ_b is the calculated bulk positron lifetime, assuming a two-state trapping model.

vacancy clusters. Above 550 K, τ_2 decreases down to a value of 150 ps for anneals in the range 700–783 K. After annealing at $T \geqslant 800$ K, this second spectral component cannot be resolved. As Fig. 1(c) shows the positron lifetime recovery is accomplished in two stages.

The onset of the first stage appears at $\simeq$500 K coinciding with the decrease of the τ_2 value and the increase of the intensity of this component, I_2, see Figs. 1(a) and (b). This means that this recovery stage is initiated by the dissolution of three-dimensional vacancy clusters. It finishes after annealing at $\simeq$700 K, giving rise to a second spectral component characterized by a lifetime of 150 ps. The second recovery stage starts after annealing at $\simeq$900 K, recovering the bulk lifetime value of 124 ps for anneals at $T \geqslant 923$ K.

3.2. V–0.3Ti and V–1Ti

The lifetime spectra of 50% rolled samples of these alloys are analyzed in terms of two exponential components without any constraint except for the final anneals. The recovery characteristics for both alloys are qualitatively similar as Figs. 2 and 3 show. The recovery of the mean positron lifetime $\bar{\tau}$ is accomplished in two stages. Just like it occurs for pure V, the first recovery stage, over the range 500–740 K, is accompanied by the dissolution of three-dimensional vacancy clusters (microvoids) as the evolution of the second lifetime component indicates. The lifetime τ_2 decreases from typical values for positrons trapped in these clusters, i.e. $\simeq$230 ps in V–0.3Ti or $\simeq$290 ps in V–1Ti, to values of $\simeq$150

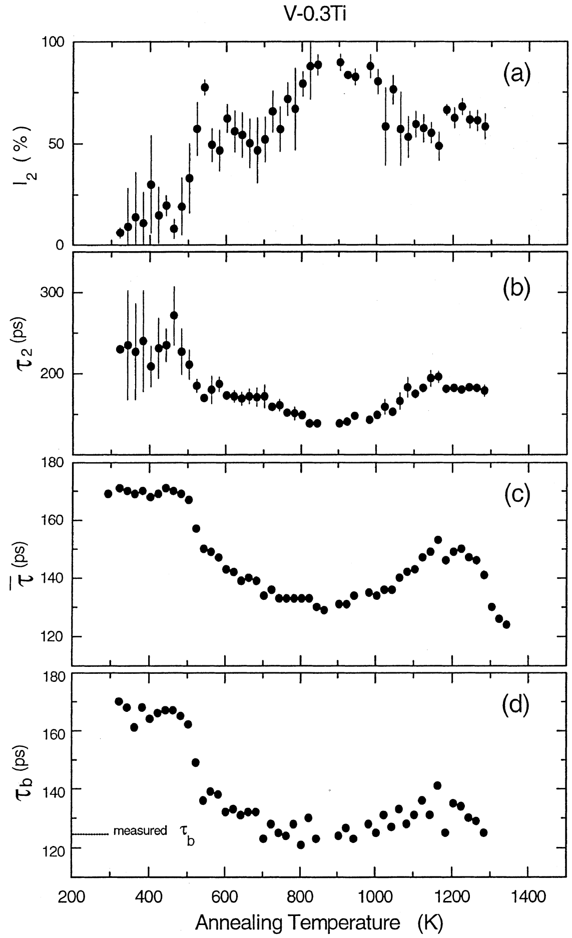

Fig. 2. Positron annihilation parameters versus annealing temperature for 50% cold-rolled V–03Ti. I_2 and τ_2 are the intensity and the lifetime of the second spectral component, respectively, $\bar{\tau}$ is the mean positron lifetime and τ_b is the calculated bulk positron lifetime, assuming a two-state trapping model.

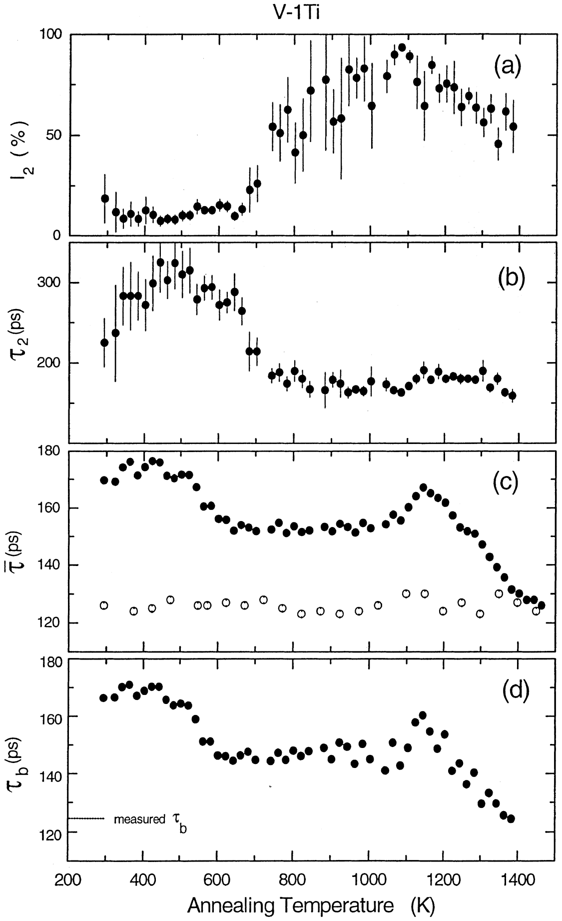

Fig. 3. Positron annihilation parameters versus annealing temperature for 50% cold-rolled V–1Ti. I_2 and τ_2 are the intensity and the lifetime of the second spectral component, respectively, $\bar{\tau}$ is the mean positron lifetime and τ_b is the calculated bulk positron lifetime, assuming a two-state trapping model. Open circles present positron lifetime measurements of the undeformed V–1Ti alloy.

and ≃170 ps, respectively, see Fig. 2(b) and Fig. 3(b). A plateau between 740 and 1040 K is observed after this first stage in the recovery curve of $\bar{\tau}$ (Fig. 2(c) and Fig. 3(c)); the $\bar{\tau}$ values remain constant at (133 ± 3) ps and (153 ± 2) ps for V–0.3Ti and V–1Ti, respectively. Likewise τ_2 values stay practically constant in the range 740–1040 K.

The second recovery stage starts at ≃1150 K in both alloys, apparently. It is preceded by a noticeably increase of the $\bar{\tau}$ value as a result of the significative increase of the τ_2 values, see Figs. 2 and 3. For anneals above 1150 K the τ_2 values stay practically constant at (183 ± 6) ps for both alloys, while the intensity I_2 goes down. The second lifetime component disappears after annealing at 1303 and 1403 K for V–0.3Ti and V–1Ti, respectively. The bulk lifetime value for these alloys, 126 ps, is finally recovered.

Figs. 4 and 5 depict the lineshape parameters S as a function of temperature for Doppler-broadening measurements performed on the samples in as-rolled state and after some annealing steps. A temperature dependence of the parameter S is found. After annealing below ≃550 K, the temperature dependence of S appears to be the same as the one found for the samples in as-rolled state. Anneals above ≃700 K produce a significative change in the temperature dependence of S; the average increasing rate $\Delta S/\Delta T$ decreases. This rate is significatively lower for V–0.3Ti.

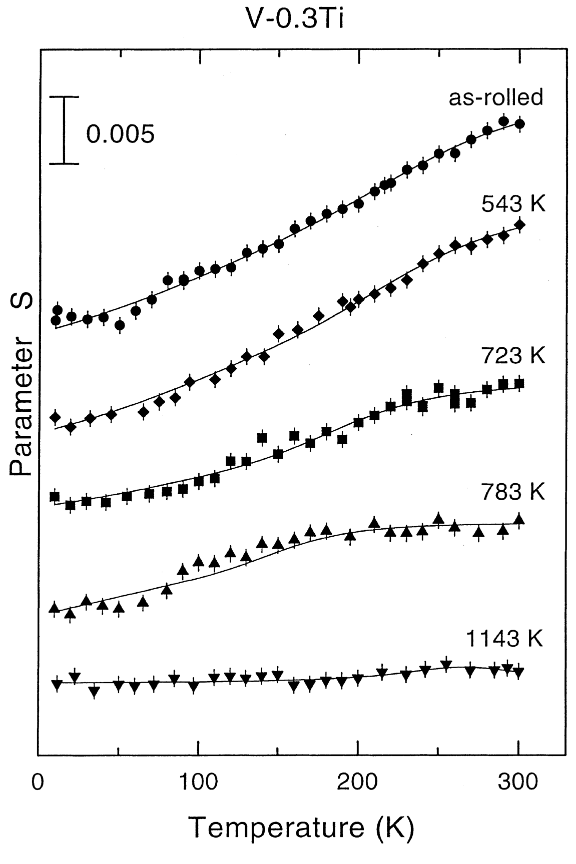

Fig. 4. Lineshape parameter S as a function of temperature for 50% cold-rolled V–03Ti after annealing at different temperatures.

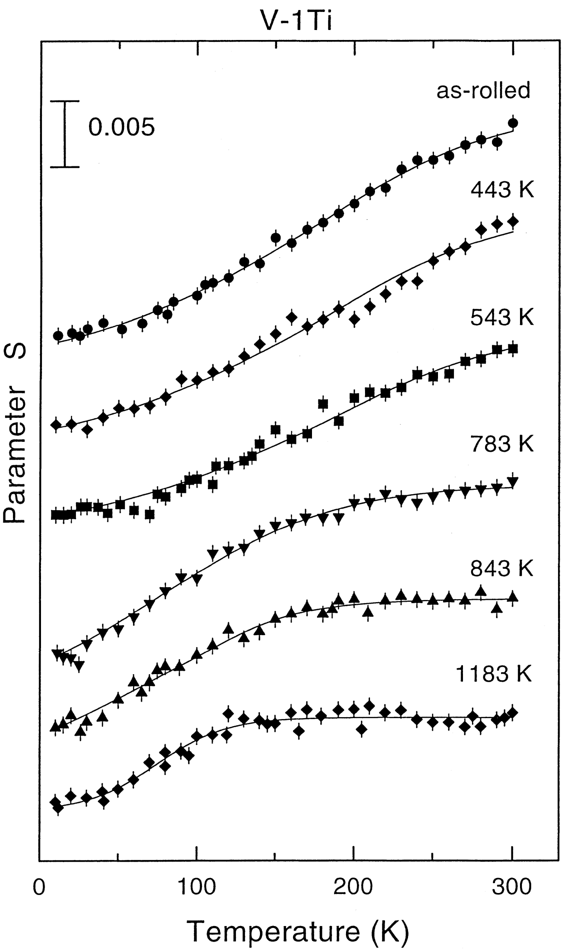

Fig. 5. Lineshape parameter S as a function of temperature for 50% cold-rolled V–1Ti after annealing at different temperatures.

The isochronal annealing experiments performed on undeformed (solution-annealed) V–1Ti samples reveal no changes in the positron lifetime; it is constant at (126 ± 3) ps after annealing in the range 900–1500 K, as shown in Fig. 3 (c).

3.3. V–4.5Ti

In opposition to the results in pure V, V–0.3Ti and V–1Ti, no evidence for three-dimensional vacancy clusters is found in 50% cold-rolled V–4.5Ti. The lifetime spectrum of these samples is single-component all over the recovery process. Fig. 6 depicts the positron lifetime

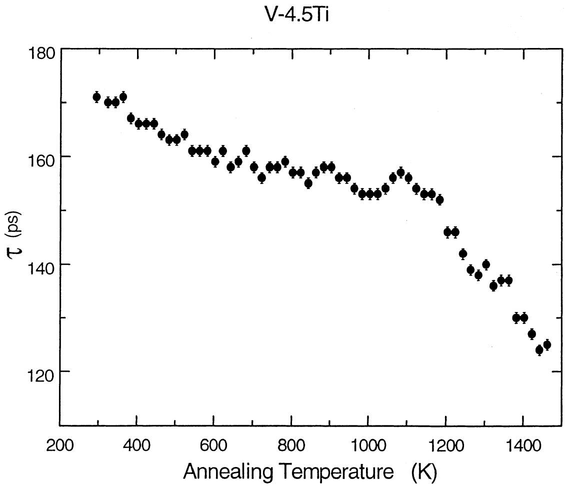

Fig. 6. Positron lifetime versus annealing temperature for 50% cold-rolled V–4.5Ti.

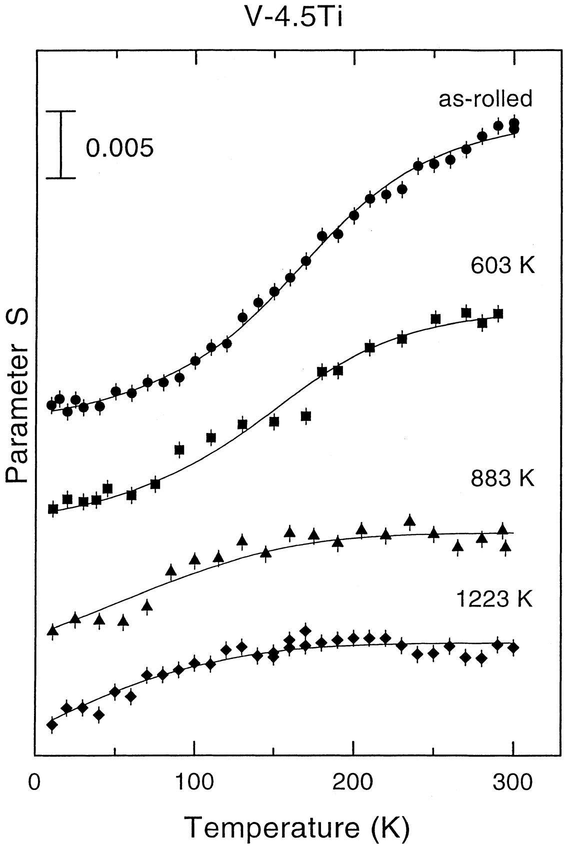

Fig. 7. Lineshape parameter S as a function of temperature for 50% cold-rolled V–4.5Ti after annealing at different temperatures.

recovery as a function of annealing temperature. The recovery curve exhibits two stages. The first stage, starting at ≃400 K, continues up to around 1000 K. The lifetime decreases continuously and slowly through this stage. The second recovery stage appears at ≃1100 K. In contrast to V–0.3Ti and V–1Ti, there is no clear evidence for a lifetime increase preceding this recovery stage.

The temperature dependence of the annihilation parameter S for as-rolled samples of this alloy is stronger than those found for V–0.3Ti and V–1Ti, see Fig. 7. The average increasing rate $\Delta S/\Delta T$ decreases after annealing above ≃600 K.

4. Discussion

4.1. Pure V

In 50% rolled samples of polycrystalline V the release and clustering of deformation-induced vacancies, revealed by the appearance of a second lifetime of ≃250 ps, is observed at ≃400 K, such as it occurs for 45% rolled single-crystals of 99.99% pure V [15]. However, the onset of the $\bar{\tau}$ recovery for polycrystalline samples appears at temperatures significatively higher than the one found for single-crystals, i.e. 500 K against ≃400 K, see Fig. 1. Moreover, the evolution of the second lifetime τ_2 during recovery differs qualitatively from that found for single-crystals [15]. τ_2 decreases to a value of 150 ps at the end of the first recovery stage instead of increase.

After annealing above 600 K, the bulk positron lifetime $\tau_b = (I_1/\tau_1 + I_2/\tau_2)^{-1}$ calculated applying the standard two-state trapping model is consistent with the measured bulk lifetime as Fig. 1(d) shows. This indicates the presence of a single positron trap having a lifetime of 150 ps at the end of the first recovery stage. These traps disappear during the second recovery stage. The disagreement between the measured τ_b and the one calculated after annealing below ⩽600 K indicates that, in addition to microvoids with a positron lifetime of ≃250 ps, other types of positron traps exist; these can be isolated vacancy–interstitial impurity pairs, dislocations and vacancy-like defects associated with dislocations such as jogs and trapped vacancy–impurity pairs [15]. In principle, we cannot elucidate whether the defects responsible for the positron lifetime of 150 ps are created by rolling or by annealing during the first recovery stage. This lifetime component has not been observed during the recovery of cold-rolled 99.99% pure V [15]. Therefore, traps responsible for the 150 ps positron lifetime appear to be associated with residual impurities. Interstitial C and O in V become mobile at ≃450 K migrating during the recovery to defects induced by deformation or irradiation [20,21]. Transmission electron microscopy (TEM) observations have revealed the formation of fine precipitates in neutron-irradiated pure V which appear to be associated to impurity segregation on defects [9,22]. Since fcc metal carbides can precipitate in V with C content as low as 71 ppm [23] and the lifetime of 150 ps is in very good agreement with that of 149 ps calculated for positrons trapped at a metal vacancy of the carbide V_3C_4 [16], we could tentatively attribute the positron lifetime of 150 ps appearing at the end of the first recovery stage to this type of V carbide. About the existence of metastable carbides and nitrides with metal vacancy excess such as V_3C_4, Ti_3C_4 and $Ti_{31}C_{32}$, see Ref.[16] and references therein.

We cannot rule out the presence of other precipitates acting as positron traps. For instance, precipitation of fine VO_y particles can also be produced during recovery since the free energy of formation for fcc VO_y is almost one order of magnitude lower than the one for VC_y [24]. Precipitates, identified as fcc VO, have been observed by TEM in pure V [25]. However, the observed positron lifetime of 150 ps is very different from values reported for VO_y. The positron lifetime for VO_y is expected to be between 199 and 214 ps when y ranges between 0.93 and 1.14 [26].

Since VC_y, VO_y and VN_y are isostructural, very miscible with each other, and have very similar lattice parameters, other plausible defects responsible for the observed lifetime of 150 ps can be V(C,O,N) precipitates. In fact, the precipitation of V(C,O,N) fine particles have been observed in annealed weld zones of V alloys [27]; the formation of these precipitates appears to be related to the existence of a high density of dislocations.The second recovery stage starting at ≃900 K, not observed in 45% rolled single-crystals [15], could be attributed to recrystallization, in principle. Nevertheless, the recrystallization temperatures reported for pure V are above ≃1023 K [28]. Therefore, something should occur to the precipitates in order that they fail to trap positrons after annealing above 900 K. Probably, their stoichiometry composition changes by solvent diffusion enhanced by pipe-diffusion along dislocations. It should be noted that precipitates with compositions VC and V_4C_3 (also VO and V_4O_3) are unable to trap positrons according to the positron affinity calculations [16,29].

4.2. V–Ti alloys

The presence of a second lifetime component of ≃250 ps in as-rolled V–0.3Ti and V–1Ti indicates that a fraction of deformation-induced vacancies coalesces in three-dimensional clusters during cold-rolling. The absence of this lifetime in as-rolled pure V and V–4.5Ti shows that (1) the Ti solute acts as a very effective trap for mobile vacancies during deformation and (2) a Ti content of 4.5% provides vacancy traps sufficient to inhibit microvoid formation during deformation. The absence of microvoids in cold-rolled pure V annealed at $T \leqslant 400$ K is attributed to the capability of the residual interstitial impurities (O, C and N) and dislocations to bind deformation-induced vacancies. For V there exists evidence for vacancy trapping by residual interstitial impurities giving rise to vacancy-interstitial impurity complexes which are dissociated at $T \geqslant 400$ K [14,20,21,30]. The observed single positron lifetime in cold-rolled V annealed at $T \leqslant 400$ K is an average lifetime due to competing positron trapping at dislocations and vacancy-interstitial impurity complexes [15]. Substitutional Ti in V is a very efficient scavenger of O, C and N [30,31]. This Ti scavenging effect sweeps interstitial impurities in the solid solution eliminating traps for deformation-induced vacancies and giving rise to a supersaturated concentration of mobile vacancies which coalesce into microvoids for Ti contents ⩽ 1 at.%. It appears that a Ti content of 4.5 at.% provides a very high concentration of vacancy traps sufficient to inhibit supersaturation of mobile vacancies during cold-rolling impeding their coalescence into microvoids.

In cold-rolled V–0.3Ti and V–1Ti, microvoids are dissolved during the first recovery stage giving rise to the appearance of defects characterized by lifetimes of (148 ± 8) ps in V–0.3Ti, and (172 ± 8) ps in V–1Ti. These values are the average τ_2 values in the interval 740–1000 K in which $\bar{\tau}$ and τ_2 stay constant, see Figs. 2 and 3. It should be noted that the opposite occurs in electron-irradiated V–0.3Ti, V–1Ti and V–4.5Ti, i.e. microvoids are formed after annealing above ≃500 K [14].

The $\bar{\tau}$ recovery curve for V–0.3Ti and V–1Ti shows a behaviour similar to that found for Ti-stabilized austenitic steel [18]. The second recovery stage in this cold-rolled steel is preceded by a remarkable increase of the positron lifetime attributed to precipitation of fine TiC particles. TEM observations have shown that the nucleation and growth of these precipitates are induced by moving dislocations during recovery [32,33]. The possibility for precipitation of Ti oxides, carbides and oxycarbonitrides in V–Ti alloys [11,23,25,27,34,35], along with our results for undeformed V–1Ti showing that isochronal annealing up to 1400 K induces no changes in the positron lifetime, indicates that the positron traps appearing at the end of the first recovery stage are related to precipitation of Ti-rich particles. Precipitation of fine Ti-rich particles, sometimes identified as fcc TiO or Ti(O,C,N), TiO_2, Ti_2O and α-Ti [10,11,22] has been observed in neutron-irradiated V–Ti alloys. Although the composition and stoichiometry of these fine precipitates cannot be determined with precision, it is well established that they are Ti-rich appearing when the irradiation temperature is above 550 K [34,35].

The change in the temperature dependence of the parameter S for cold-rolled V–0.3Ti and V–1Ti coincides with the end of the first recovery stage of $\bar{\tau}$. It has been shown that the temperature dependence of the parameter S for V is controlled by the trapping rate for deep traps, i.e. vacancy clusters, vacancy–impurity complexes and single dislocation jogs, as well as by the ratio between the trapping rates for dislocations at 0 K and the trapping rate for deep traps [15]. Thus, these changes in the temperature dependence of S appear to be due to the disappearance of some sort of vacancy–impurity complexes and dislocation jogs, in addition to vacancy clusters, rather than dislocations. During this recovery stage vacancies, interstitial impurities and the Ti solute, assisted by dislocation climb, must sink into nuclei for Ti-rich precipitates. The interaction of Ti atoms with deformation-induced defects can provide strong driving forces for their diffusion and segregation on climbing dislocations. Because of the high precipitate/matrix mismatch expected in the V–Ti alloys, i.e. 30–42% for oxides and carbides, the mechanism of repeated precipitation on climbing dislocations would be operative [36]. This is the mechanism responsible for carbide precipitation in austenitic steels.

Assuming the formation of Ti-rich precipitates, and according to calculations of positron lifetimes and affinities in metal carbides, nitrides and sulfides [16,17], vacancies in the lattice of non-stoichiometric precipitates would be the traps responsible for the positron lifetime observed at the end of the first recovery stage. The plateaus in the recovery curves of $\bar{\tau}$ and τ_2 for annealing temperatures between 740 and 1040 K, accompanied by an increase of I_2, indicate that precipitation occurs through this temperature interval, see Figs. 2 and 3.

For V–0.3Ti the calculated τ_b agrees with the measured one indicating that the τ_2 value of (148 ± 8) ps found after annealing over the range 740–1000 K is the positron lifetime for defects resulting from the first recovery stage. This lifetime value coincides with the bulk lifetime for α-Ti (147 ps) as well as with the calculated lifetime for positron trapped at a metal vacancy of the metastable non-stochiometric carbide V_3C_4 (149 ps) [16]. Also, it agrees with the calculated positron lifetimes for C vacancy clusters in Ti carbides: 147 and 153 ps for 3 and 4 vacancies, respectively [16].

For V–1Ti, the τ_2 value of (172 ± 8) ps found following the first recovery is in very good agreement with the calculated lifetime for positrons trapped at the Ti vacancy in a $Ti_{31}C_{32}$ supercell, 174 ps [16]. In contrast with the observation in pure V and V–0.3Ti, the calculated τ_b values for V–1Ti continue to be inconsistent with a two-state trapping model after the first recovery stage, indicating the presence of other positron traps besides the 172 ps traps.

The recovery of cold-rolled V–4.5Ti starts with a decrease of the positron lifetime from 170 to 157 ps between 380 and 700 K remaining constant in (157 ± 2) ps for annealing temperatures up to ≃940 K. This value is very close to the measured positron lifetime of (160 ± 2) ps for TiC [37,38] that agrees with the one calculated for the Ti vacancy in the Ti_3C_4 carbide, 161 ps [16]. Moreover, non-stoichiometric carbides such as $Ti_{31}C_{32}$, Ti_3C_4 and V_3C_4 have much higher positron affinities than V and Ti , i.e. between −7.9 and −6.6 eV against −3.4 and −4.1 eV for V and Ti, respectively [16,17]. Therefore, when particles of these carbides precipitate in a V matrix, positrons can be trapped in these particles annihilating there. If their concentration is very high positrons will annihilate only in these particles.

The measured positron lifetime for $TiO_{0.86}$ and $TiO_{1.14}$ are 203 and 200 ps, respectively [26]. These values are very different from the found τ_2 values which appear to be related to precipitates. All above results compel us to attribute the lifetime observed at the end of the first recovery stage to precipitation of Ti-rich particles, likely Ti carbides or Ti(C,O,N). In pure V and V–0.3Ti only one type of precipitates responsible for positron trapping exists, as discussed above. In V–1Ti, after finishing the first recovery stage more than one type of positron traps exist, suggesting the presence of more than one type of precipitates. The absence of the $\bar{\tau}$ increase preceding the second recovery stage in pure V points to the Ti-rich precipitates as the defects responsible for this increase in the recovery curve for V–0.3Ti and V–1Ti. Vacancies released during the recrystallization process should sink into these Ti-rich precipitates becoming incorporated in their lattices. This can give rise to vacancy clusters in the precipitate lattice. These clusters would give a positron lifetime a bit longer than the one for a single vacancy. The light increase of τ_2 and τ_1 found at the onset of the second recovery stage for V–0.3Ti and V–1Ti supports the above. Also, other interpretations to the $\bar{\tau}$ increase may be given. For instance, vacancies released during recrystallization become trapped at Ti rich precipitate/matrix interfaces. It should be noted that the τ_2 value is (183 ± 6) ps for anneals above 1150 K; this value coincides with the positron lifetime for vacancies in V [14]. For V–4.5Ti a significative increase of $\bar{\tau}$ is not observed, likely due to positron trapping saturation.

The $\bar{\tau}$ recovery after annealing above 1150 K is attributed to a gradual vacancy sweep, either in the precipitates or at the precipitate/matrix interfaces, which is induced by precipitate coarsening following recrystallization. Recrystallization in V–Ti is expected to occur between 1100–1200 K [28,39]. Scanning electron microscopy observations performed on annealed samples showed that grain growth occurred for annealing temperatures around 1300 K. It should be noted that this second recovery stage coincides with the annealing-induced suppression of helium trapping at unidentified fine precipitates that are observed in V–4Cr–4Ti [40].

5. Conclusions

Ti solute concentrations of about 4.5 at.% in V inhibit the coalescence of deformation-induced vacancies into microvoids. The results indicate the formation of precipitates during the first recovery stage of cold-rolled V–Ti alloys and 99.9% pure V. The precipitation appears to be induced by dislocations. The second recovery stage, found at temperatures above ≃1150 K for V–Ti alloys, is attributed to the annealing of vacancies associated to the precipitates. This annealing is induced by the precipitate coarsening following recrystallization.

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