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X-ray diffraction analysis of titanium tritide film during 1600 days

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ABSTRACT

The generation and accumulation of ^{3}He by tritium decay modified the physical and chemical properties of tritides. Here the evolution of lattice defects in long-aged titanium tritide films is investigated by X-ray diffraction and changes in the positions, intensities and line shapes of diffraction peaks have been determined over a period of about 1600 days (>4 years). Texture effects are also observed by biased intensities in standard θ–2θ scans. The results show that the $TiT_{1.5}$ film keeps an fcc structure during 1600 days and reveals an *hkl*-dependent unit-cell expansion and line width broadening which are interpreted in terms of isolated tetrahedral interstitial ^{3}He atoms and isolated bubble growth models by dislocation loop-punching or dislocation dipole expansion combined with Krivoglaz theory. In the first 12 days of aging, isolated tetrahedral interstitial ^{3}He atoms or ^{3}He clusters are formed, then interstitial ^{3}He atoms diffuse into (1 1 1) planes and precipitate into clusters. The spontaneous formation of Frenkel pairs, the self-interstitial atoms produced are built into dislocations resulting in formation platelet bubbles and dislocation dipoles between 12 and 27 days. Above 27 days, multiple stages of ^{3}He bubbles growth appear: (1) between 27 and 85 days platelet helium bubbles growth by dislocation dipoles expansion, (2) between 85 and 231 days the transition from platelet bubbles to sphere bubbles by loop emission, (3) after 231 days sphere bubbles growth by dislocation loop-punching and probably formation of sub-grain boundaries by dislocation rearrangement.

1. Introduction

Tritium is a hydrogen isotope of considerable interest and has important technological applications, especially in the nuclear industry. However, the radioactive nature of tritium imposes many conditions on its handling and storage. It has been recognized for a long time that the best way to store hydrogen is in the form of a hydride, which has the advantage of safety, easy recovery, and also much larger quantities of hydrogen can be stored per unit volume than in its liquid form. Therefore, it is quite natural to propose the storage of tritium in the form of a tritide, and metals such as palladium, titanium, zirconium, erbium, uranium, and the intermetallic alloys such as lanthanum–nickel and zirconium–cobalt, are commonly used for this purpose.

Since tritium decays into ^{3}He with a half-life of 12.3 years, through the decay process $T \rightarrow {}^3He + \beta^- + \nu^-$, where β^- is an electron which emitted with 18.582 keV of energy, and ν^- is an anti-neutrino, metal tritides are time-dependent ternary systems in which the increase in the ^{3}He concentration corresponds to the decrease in the tritium concentration. The recoil energy of ^{3}He in this decay process is quite small, ~1.03 eV, and is insufficient to cause any damage to the lattice [1]. The decayed helium stays in the solid and does not release until a critical helium concentration has been reached. This ^{3}He is highly insoluble and immediately starts to aggregate into bubbles inside the tritide material as has been described in detail. The bubbles and dissolved He are thought to induce strain in the metal, which induces large structural and microstructural changes in the host lattice, leading to modifications of its thermodynamical properties with aging.

Concerning titanium tritide only few works are available in the literature and deal mainly with thermal desorption [2–4], TEM observations of helium bubbles [5,6] and measurements of the amount of swelling [7]. The complicated handling of tritium due to safety considerations has limited the structural characterization of aged titanium tritide, but prior work on the time dependence of structural properties in titanium tritide and tritide-deuteride powders has been reported [8–10]. Tritium is stored in a metallic thin-film target in neutron generators: this is the case of titanium, zirconium, hafnium, erbium, scandium, yttrium, etc. [9,11]. The aging behavior of tritide film may be influenced by the film substrate, because the grain orientation (texture) of film can be dictated by the substrate and the film geometry will limit the expanding of grains. It is of interest to examine titanium tritide films and compare its behavior to that of titanium tritide powders and other tritide films. In the present work, we focus our X-ray diffraction (XRD) analysis on an aged titanium tritide film. We present the first results of XRD patterns of titanium tritide film after aging for 1600 days. The

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relationship between the amount of decayed ^{3}He, aging time and lattice damage are discussed.

2. Experimental

Titanium films were deposited by resistively-heated evaporation onto rolled-molybdenum substrates to nominal thicknesses of 3 μm. The titanium films were removed from the evaporator at ambient conditions and then placed in a special tritiding apparatus.

According to the Ti-H phase diagram, at room temperature, the α-Ti phase (hcp Ti with interstitially distributed H atoms) is stable for a H content less than 0.14 at.%, followed by a mixed $\alpha + \delta$ phase for H/Ti ratio less than 1.5 and finally by a single phase (δ-fcc CaF_2-like structure of titanium hydride which contains four titanium atoms forming an fcc lattice with the tritium atoms occupying the tetrahedral sites) for a H/Ti ratio between 1.5 and 2, especially with stoichiometric defects (at most 25% vacancy of tetrahedral site in fcc lattice). The stoichiometric defects may enhance trapping by acting as an initial nucleus for helium. The samples were loaded with high-purity tritium gas to produce a tritide with stoichiometry close to $TiT_{1.5}$. The samples were then stored at room temperature in a sealed vacuum envelope until they were studied by XRD.

The XRD measurements were performed with a Philip X'Pert Pro diffractometer using Cu Kα radiation, research was done at room temperature every three days during the first 2 weeks, every two weeks during the first 3 months and every month for larger aging time. Each pattern was registered from 30° to 90° with a step of 0.05° in 2θ and were indexed in a face centered cubic structure. The (1 1 1) and (3 1 1) peaks of $TiT_{1.5}$ were scanned with higher resolution (0.01° step).

3. Results

The observed (1 1 1) peaks and (3 1 1) peaks of $TiT_{1.5}$ (space group Fm-3 m) aged for 8 days, 85 days, 562 days and 1519 days after T charging are shown in Fig. 1, respectively. It reveals three main changes occurring during aging: (1) a peak shift, (2) a peak broadening with an associated decrease in the maximum intensity, and (3) an asymmetry towards higher angles. The results show the (1 1 1) diffraction peak and (3 1 1) diffraction peak shift towards smaller angles corresponding to an increase of the average lattice parameter during the first 231 days and the peaks shift towards larger angles corresponding to a decrease of the lattice parameter after 231 days. After only 20 days of storage, noticeable peak broadenings appear and after 198 day, the width of the peak is significantly modified. These peak changes are generally attributed to helium bubble strains and crystal defects due to the continuous decay of tritium and build up of He.

The generation of helium (tritium decay) up to the ratio of 0.36 (He_{gen}/Ti corresponding to the number of generated helium atom by metal atom) does not induce α-phase of titanium, in agreement with the results of titanium tritide powders [8–10].

The relative lattice parameter change in $TiT_{1.5}$ film versus the helium content (He_{gen}/Ti) sample are presented in Fig. 2. The value of lattice parameter was calculated from the position of the diffraction maxima (1 1 1) and (3 1 1) center of gravity.

Previous studies [8] have revealed an *hkl*-independent unit-cell expansion and found unchanged lattice parameter of $TiT_{1.9}$ powders at helium storage up to the ratio of $He_{gen}/Ti \approx 0.09$, quite a sharp decrease of the lattice parameter in the range of $He_{gen}/Ti = 0.09–0.12$ and a linear decrease of the fcc-period takes place in the range, corresponding to the He_{gen}/Ti from 0.12 to 0.3. On the contrary, an *hkl*-dependent unit-cell expansion was found in

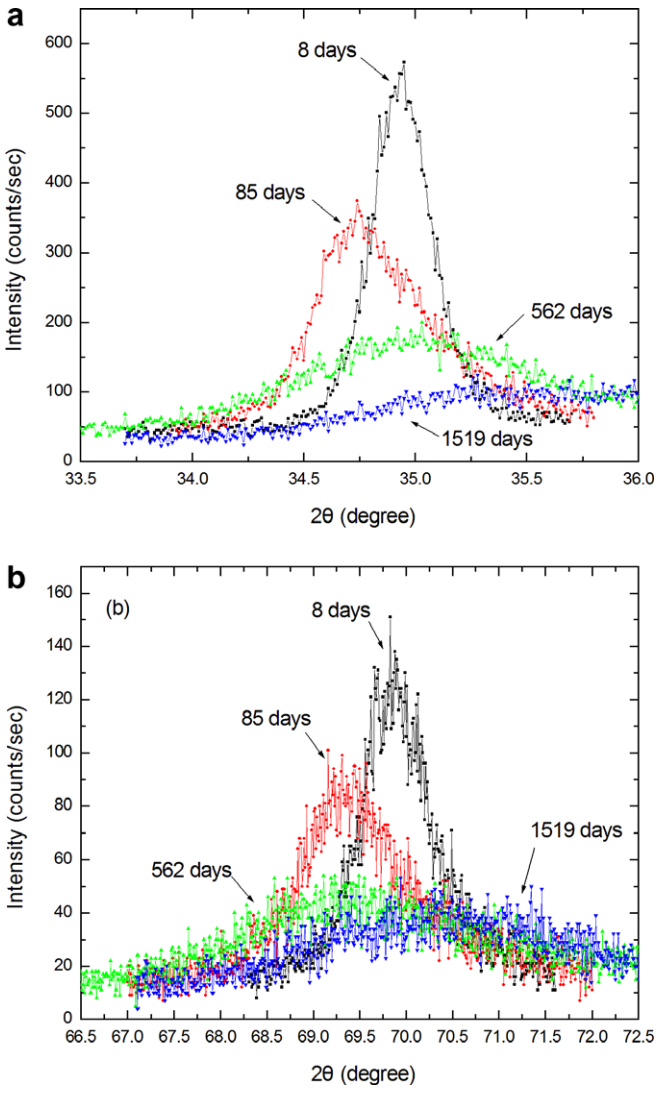

Fig. 1. Diffraction peaks of the titanium tritide film after different aging times: (a) (1 1 1) peak and (b) (3 1 1) peak.

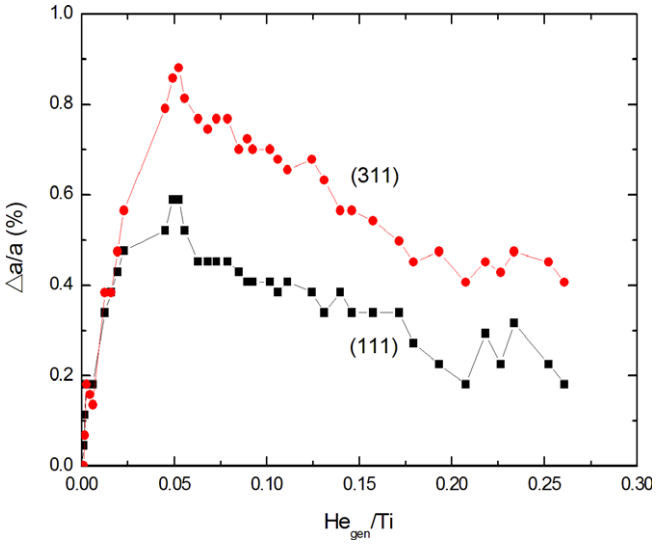

Fig. 2. Relative change of the lattice parameter a in the $TiT_{1.5}$ film.

this study. We observed, in agreement with the work of Vedeneev et al. [9] and Flament [10], that the lattice parameter of $TiT_{1.5}$ film continuously increase versus the helium content during aging 231 days ($He_{gen}/Ti = 0.052$) and then slowly decrease after 231 days. The increase of lattice parameter versus helium content shows three domains (Fig. 2). (1) The first 12 days ($He_{gen}/Ti = 0.003$), the lattice parameter increases linearly with $d(\Delta a/a)/dc_{He} = 0.80$ (1.85×10^{-4}/day), (2) between 12 and 27 days ($He_{gen}/Ti = 0.006$), the (1 1 1) lattice parameter does not change and the (3 1 1) lattice decreases little, (3) after 27 days, the lattice swelling progressively slows down and a considerable divergence of lattice period values is observed in which the (3 1 1) grains expand out-of-plane more than (1 1 1) out-of-plane grains indicating an *hkl*-dependent unit-cell expansion. The results clearly show the lattice distortion exhibit anisotropic characteristics and it is difficult to accurately calculate the lattice constants of long-aged $TiT_{1.5}$ film from its XRD patterns.

The relative line half-width broadening (B) of (1 1 1) and (3 3 1) for titanium tritide film versus generated helium content is given in Fig. 3. First, the line width of titanium tritide film remains practically constant before 12 days ($He_{gen}/Ti = 0.003$). After 12 days, the line-width increases over the entire range of He content examined and also shows anisotropic behavior for the reflections lying on (1 1 1) and (3 1 1). In the (1 1 1) plane the observed rate of broadening of the line width is at a lower level than in the (3 1 1) plane. The increase of line width versus helium content shows three domains (Fig. 3): (1) the first 85 days ($He_{gen}/Ti = 0.020$), the line-width increases slowly with helium content, (2) between 85 and 231 days ($He_{gen}/Ti = 0.052$), the line-width increases steeply, (3) after 231 days, the line width progressively slows down.

4. Discussion

In the tritide case, ^3He atoms are generated in the materials itself. This is a 'gentle' method in which the helium is introduced in a well controlled and understood process and the helium spatial distribution is uniform as long as the tritium is uniformly distributed. The helium is introduced without significant damage to the metal lattice, as compared to implantation or nuclear reaction cause significant metal lattice damage occurs. In addition the metal tritide is thermodynamically stable.

Since the ^3He atoms are created by tritium decay with insufficient energy to displace the metal host atoms, they occupy interstitial sites and are mobile [12,13] at room temperature. There are two interstitial accessible to ^3He atoms. One is the tetrahedral interstitial site left by the decaying tritium; the other is the octahedral interstitial site in the center of a face centered cube. Whereas ^3He, as with the other inert gases, has solubility limited to the sub-ppm range. Therefore, ^3He created on tritium transmutation in metals has a strong tendency to precipitate into clusters. When the number of ^3He interstitial atoms in a cluster exceeds a critical value, small ^3He clusters produce enormous lattice distortions and lead to the formation of near-Frenkel pairs (lattice atoms pushed into nearby interstitial sites), and the self-interstitial atoms (SIA's), pushed from their lattice sites, themselves cluster on one side of the helium bubble to reduce the strain energy [14]. Above a given size of ^3He clusters, dislocation loops form from SIA's bound to the resulting He-vacancy clusters or bubbles, respectively [15,16]. The continuous formation of dislocation loops or dislocation dipoles leads to the creation of a network of dislocations and possibly the formation of sub-grain boundaries by dislocation rearrangement [17].

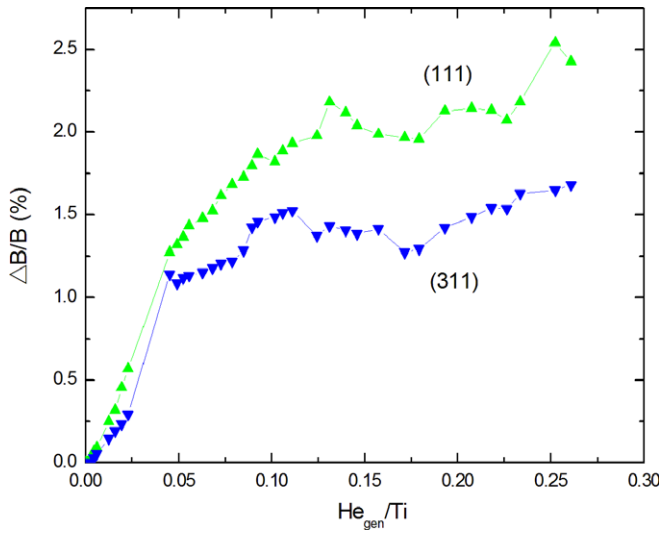

Fig. 3. Relative change of the diffraction peak half-width B of the $TiT_{1.5}$ film.

According to Krivoglaz [18], if lattice defects of finite size (isolated interstitials, dislocation loops, and bubbles) reside in the lattice as single point defects or within isolated clusters, these processes result in lattice expansion and distortion fields that manifesting themselves in peak shifts towards small angles with diffuse scattering around the peak, but have no effect on the line width. On the other hand, if SIA's are incorporated into defects of infinite size such as dislocations and dislocation networks, they induce a broadening of Bragg reflections or Debye–Scherrer lines.

Provided that every decayed ^3He occupies the tetrahedral interstitial site or the octahedral interstitial site during the first few days following tritium introduction into the metal, relaxations around a interstitial ^3He can be express in terms of vector displacements of neighboring atoms surrounding the defect. These displacements may be related to lattice parameters change of the material using continuum elasticity theory [19]. In addition, in a homogeneous area of titanium tritides [8–10], one can expect that the decrease of tritium concentration in tritides due to tritium decay must cause a linear decrease of the lattice parameters with $d(\Delta a/a)/dc_T = 2.26 \times 10^{-2}$. This is about 3% of the total expansion measured for $TiT_{1.5}$ films during the first 12 day. With a hard sphere model [20], the lattice parameter change of the sample film ($d(\Delta a/a)/dt$) calculated for an interstitial ^3He atom in a tetrahedral interstitial site is about 1.9×10^{-4}/day which agrees quite well with our experimental result during the first 12 day. At the same time, the line width of the sample remains constant. Therefore the film sample changes during the first 12 day can be explained by ^3He atoms produced from the radioactive decay of tritium. Although not soluble in the tritide, the He is apparently retained in a metastable state, in surprisingly large quantities at the tetrahedral interstitial sites where it is born. This result is in accord with the results of electronic structure calculations [21,22].

The stable (1 1 1) lattice parameter and the minor (3 1 1) lattice contraction between 12 and 27 days can be related to additional ^3He atoms that begin to lower energy sites which are those with lower electronic density such as vacancy-like defects formed by 'self-trapping' mechanism as discussed above and accumulate to form bubbles. However, for this mechanism the number of ^3He atoms at one defect-free nucleation site must exceed a certain value. This kind of nucleation site is always with relatively large free space. As is well known, the lower indices planes are associated with larger plane distances. In fcc structures, the (1 1 1) planes provide the greatest "space" for interstitial precipitates. So we conclude that during this aging time interstitial ^3He atoms prefer to accumulate in the (1 1 1) planes and form ^3He bubbles.

According to the dislocation loop-punching mechanism and the dislocation dipole expansion mechanism [23,24], in which the

number density of ^{3}He bubbles is assumed to be 5×10^{23} bubbles/m^3 and s (the thickness of platelet bubbles) is presumed to be 2.5 d_{111} (the distance between (1 1 1) planes), the ^{3}He bubble of platelets shape appear to be the preferred shape rather than spherical shape in titanium tritide when the major radius of the bubbles is less than 3.5 nm (He/Ti = 0.032). Tensile stress created by the spherical bubbles and the platelets bubbles both produce a positive $d(\Delta a/a)/dc_{He}$. Fig. 4 shows relative change of the lattice parameters which are corrected for the corresponding decrease of the average lattice parameters due to tritium decay ($(\Delta a/a)_{tot} = (\Delta a/a)_{obs} - (\Delta a/a)_T$) and compared with the computed change of the lattice parameter da/a, in which the number density of ^{3}He bubbles and s are also assumed to be 5×10^{23} bubbles/m^3 and 2.5 d_{111}, respectively, due to ^{3}He bubbles with increasing He/Ti in the titanium tritide film. The lattice dilation data for $TiT_{1.5}$ film shows the existence of multiple stages of ^{3}He bubbles growth: (1) rapid lattice parameter dilation shows in accordance with platelet bubbles growth when the He/Ti less than 0.02, in agreement with TEM observations of helium bubbles [26], (2) the transition from platelet bubbles to sphere bubbles between He/Ti = 0.02 and He/Ti = 0.05, (3) slow lattice parameter dilation shows in accordance with sphere bubble growth by normal loop-punching when He/Ti greater than 0.05. A study of helium nano-bubble evolution in aging titanium tritides will be reported in a future publication.

Information on the dislocation system is provided by the width and the shape of the diffraction peak, which represent a measure for the dislocation density and an indicator for the character of the dislocation distribution, respectively [17].

Random distributions of narrow dislocation dipoles of interstitial type which could form from SIA's and remain attached to platelet bubble could account for the broadening of Debye–Scherrer peaks, and also would result in a peak shift corresponding to that of an equivalent distribution of isolated interstitial-type dislocation loops [17], in agreement with our observations during the first 85 days. The asymmetry of the diffraction peaks (more intensity at the high-angle side, Fig. 1) is expected for interstitial-type dislocation loops [25], which supports the preserve of interstitial-type dislocation dipoles.

A network of dislocations arising from the continuous formation of dislocation loops or dislocation dipoles could account for the steep line-width increases between 85 and 231 days. After 231 days, the rate of line-width increase slows down, which possibly indicates the formation of sub-grain boundaries by dislocation rearrangement. The further evolution of rocking curves of $TiT_{1.5}$ films during long aging time should be studied.

In general, the relative lattice parameter change and the relative line half-width broadening accord with Vedeneev's study [9] and Flament's study [10] except that the unit-cell expansion in our work is larger and *hkl*-dependent. However there is severe quantitative disagreement between our study and Gavrilov's study [8]. These differences are both in the relative lattice parameter change and the relative line half-width broadening. One possible explanation for these differences could lie in the lattice period of titanium tritide due to different off stochiometry compositions, initial T/Ti ratios of 1.9, 1.8 and 1.6 were chosen by Gavrilov, Vedeneev, Flament respectively. Because there are more stoichiometric defects (tetrahedral interstitial site) in the more off stochiometry composition (lower initial T/Ti ratio). These defects may enhance trapping by acting as an initial nucleus for the helium. We hypothesize that the helium concentration, located in the interstitial lattice site, is more in the lower initial T/Ti ratio titanium tritide.

The possible key reason for the *hkl*-dependent unit-cell expansion revealed in this work is the influence of the substrate, the grain orientation (texture) of film can be dictated by the substrate and the film geometry will limit the expansion of grains. For instance, the film used in this study shows texture effects that bias intensities in standard θ–2θ scans (Table 1). Pole-figures for $TiT_{1.5}$ films and the influence of the molybdenum substrates should be studied in detail to achieve a better understanding texturing effects than is possible with simple a θ–2θ scans.

Standard θ–2θ XRD analysis only measures diffraction planes that are in the plane of the film. When the ^{3}He bubble formation and growth, significant in-plane macro-strain could also exists in $TiT_{1.5}$ film, which have been revealed in ErT_2 films [27]. In films an in-plane compressive force develops because $TiT_{1.5}$ grains wish to expand but the film geometry limits this. Out-of-plane expansion is not likewise hindered. So an *hkl*-dependent unit-cell expansion can be explained in terms of ^{3}He bubble expansion along (1 1 1) planes [27] in the $TiT_{1.5}$ film, too. One could assume that one [1 1 1] direction out-of-plane expands easily and three additional [1 1 1] directions just 20° from in-plane have hindered expansion in (1 1 1) out-of-plane grains, on the other hand, for (3 1 1) out-of-plane grains one [1 1 1] direction out-of-plane just 10° from in-plane is hindered expansion and three additional [1 1 1] directions (two directions 31.5° from in-plane, one direction 61.5° from in-plane) have less internal opposition from other [1 1 1] directions which would expand easily and more uniform. Therefore (3 1 1) out-of-plane grains expand out-of-plane more than (1 1 1) out-of-plane grains and (3 1 1) peaks broaden less than (1 1 1) peaks.

However, this study not sufficiently accurate to yield a quantitative picture of the underlying lattice damage by helium production, and doses not allowed us to establish the detailed materials micro-structures effect of helium behavior in titanium tritide films. Future experiment works on the swelling, rocking curves, pole-

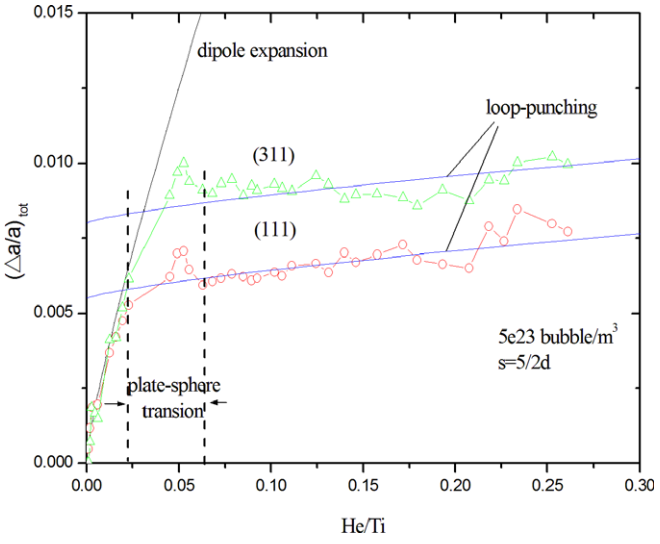

Fig. 4. Total relative change of the lattice parameter corrected for tritium decay in the $TiT_{1.5}$ film. The dashed line corresponds to the simulative lattice parameter change due to platelet helium bubbles growth by dislocation dipoles expansion. The dashed-dotted lines correspond to the simulative lattice parameter change due to sphere bubbles growth by dislocation loop-punching.

Table 1
Peak intensity of different titanium tritide *hkl* planes.

hkl	Obs. RI (%)[a]	Exp. RI (%)
1 1 1	100	100
2 0 0	42.5	205.9
2 2 0	24.7	150.1
3 1 1	23.1	31.8

[a] ASTM powder diffraction File 78–2216, $TiD_{1.5}$.

figures, thermal desorption and TEM observations of helium bubbles for the $TiT_{1.5}$ films will try to answer these questions.

5. Conclusion

Our experimental results and their interpretation may be summarized as follows.

The $TiT_{1.5}$ film maintains an fcc structure during 1600 days of aging. XRD analysis of $TiT_{1.5}$ films during aging reveals an *hkl*-dependent unit-cell expansion and an *hkl*-dependent line width broadening in which (3 1 1) grains expand out-of-plane more than (1 1 1) out-of-plane grains, but (3 1 1) peaks broaden less than (1 1 1) peaks. The texture effects are also observed by biased intensities in standard θ–2θ scans.

In the first 12 days of aging, up to about 0.3 at.% ^{3}He, the linear increases in the lattice parameter and the stable line-width can be explained by the formation of isolated tetrahedral interstitial ^{3}He atoms or ^{3}He clusters. Between 12 and 27 days, the (*hkl*)-dependent lattice parameter change and the broadening of the line width can be explained by assuming that in this stage interstitial ^{3}He atoms diffusion into (1 1 1) planes and precipitate into clusters, which then spontaneous form Frenkel pairs, and SIA's produced are built into dislocations resulting in formation platelet bubbles and dislocation dipoles. Above 27 days, both the lattice parameter increase and the broadening of the line width agree well with multiple stages of ^{3}He bubbles growth. Between 27 and 85 days, the data are consisted with platelet helium bubble growth by dislocation dipoles expansion. An apparent transition from platelet bubbles to sphere bubbles by loop emission between 85 and 231 days is followed, after 231 days by sphere bubbles growth by dislocation loop-punching and probably the formation of sub-grain boundaries by dislocation rearrangement.

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