SOI: 1.1/TAS DOI: 10.15863/TAS
International Scientific Journal
Theoretical & Applied Science

p-ISSN: 2308-4944 (print) **e-ISSN:** 2409-0085 (online)

Year: 2015 **Issue:** 04 **Volume:** 24

Published: 30.04.2015 http://T-Science.org

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SECTION 3. Nanotechnologies. Physics.

RADIATION AND ANNEALING PROCESSES IN BINARY ALLOYS OF TITANIUM

Abstract*: In this paper the results of thermal cycling influence on radiation damaged alloys of the titanium from various states are presented. It is shown that these processes called a diffused-free relaxation can lead to reorganization and crushing of grains-crystallites therefore the new condition of metal remains as hardening. It was found that radiation by charged particles leads to significant redistribution of the electron density in the defects which is manifested in the increase in the probability of annihilation WP 2-3 times and reducing the angle of the Fermi momentum. Thermal cycling of the annealed samples also causes noticeable increase of WP and reducing of θ_F , in some cases close to the effect of protons radiation.*

Key words*: thermal cycling, radiation, α - particles, positrons, binary titanium alloys.*
Language*: English*
Citation: Mukashev KM, Yar-Mukhamedova GS, Shadinova KS (2015) RADIATION AND ANNEALING PROCESSES IN BINARY ALLOYS OF TITANIUM. ISJ Theoretical & Applied Science 04 (24): 10-14.
Soi: http://s-o-i.org/1.1/TAS*04(24)2 ***Doi***: crossref http://dx.doi.org/10.15863/TAS.2015.04.24.2

Introduction

The influence of thermal effects on the characteristics of positron annihilation was established soon after the discovery of the positrons localization phenomenon in defects [1, p. 795; 2, p.2433; 3,p.422]. But despite considerable progress in the study of condensed state, the possibilities of the method of electron-positron annihilation (EPA) found some limitations, especially in the study of the characteristics of the grain boundaries of polycrystalline, which also represent defective areas containing a higher density (up to 10^8-10^{10} cm^2) dislocations. And here matter isn't that these defects aren't able to provide capture and localization of positrons and that the mean free path of the latter $l = 2\sqrt{D_+ \cdot \overline{\tau}}$ (D_+ = 0,1 cm^2/s and $\overline{\tau} = 10^{-10} s$) is about 10-5 cm that is much less than crystallite size of conventional polycrystalline. Therefore the relative share of the positrons trapped by these borders is very small and the contribution them in integrated effect is actually not notable. In these cases it is possible to do otherwise. In these cases it is possible to arrive in a different way. In paper [4, p. 75] studied thermo-cycling influence by room cooled to liquid nitrogen temperature and followed by heating to room behavior annihilation characteristics in this case - on changes of speed of the account of N(0) in a maximum of a range of the angular distribution of annihilation photons (ADAP) in time. As objects of research were chosen poly-and single crystals of Si and Zn with the original - the annealed condition. As a result of thermal cycling for monocrystals of noticeable changes of speed of the account of N(0) neither on an absolute value, nor in time wasn't revealed. For polycrystalline non-recurrent thermal cycling caused a significant increase in N (0) compared with baseline and subsequent irregular oscillations in time. The amplitude of these deviations is much higher than the statistical accuracy and they decayed over 190 minutes for Si and 260 minutes for Zn. The absence of oscillations N (0) in the spectrum of single crystals and their presence at polycrystals are the certificate of that the processes arising owing to thermal cycling in crystallites and grains in the latter case have impact on annihilation characteristics. In this regard there was a problem – whether observed processes are constantly acting or really come to the end with attenuation of an oscillation? In other words it was necessary to determine whether the observed phenomenon is associated with the new structural defects in the crystal lattice of the metal and how it affects in parameters of the annihilation radiation damaged metals.

Test methods and research results

These issues are decided to be solved on the basis of example of alloys system Ti - In of various composition. Thermal cycling test has been applied to instances with initial (annealed during 1 hour in vacuum 10^{-6} torr. under 900^0C), radiated α - by particles (E = 50 MeV, D=5·10^{15} cm^{-2}) and protons (E = 30 MeV, D=5·10^{15} cm^{-2}) condition. Experimental research of materials has been carried out by measurement of annihilation photon angular distribution (APAD).

The main purpose of any analysis, including the analysis of annihilation radiation angular distribution, is decomposition of spectrum on actual number of components with specifying relative fraction of each component, which defines one or another mechanism of positron annihilation [5, p. 308; 6, p.1572]. It is considered that APAD spectrum for most materials consists of two components - narrow parabolic and more extended Gaussian. Parabolic component results from positron annihilation by conduction electrons or valence electrons, which behavior is similar to degenerated electron gas. This distribution can be mathematically described by the following equations:

$$N_P(P) = N(0)(P_F^2 - P_Z^2) \text{ for } P_Z \le P_F$$

or

$$N_P(\theta) = N(0)(\theta_F^2 - \theta^2) \text{ for } \theta \le \theta_F ,$$

Where P_F and θ_F is Fermi momentum and its relevant angle $P_F = mc\theta_F$. Both distributions become zero beyond range of P_F and θ_F, respectively. However, if you compare theoretically-derived curve APAD for valence electrons of each specific metal with experimentally measured curve, it is easy to define their considerably outstanding difference between each other, especially beyond central parabolic section. The nature of wide-angular component of APAD is connected with interaction of valence electrons and ionic lattice, in consequence of which their condition does not meet coboundary momentum P_F and becomes smeared-out, and also with positron annihilation with ion core inner electrons, possessing the momentum which considerably exceeds the Fermi momentum. In this case APAD logic is sufficiently described by the Gaussian function:

$$N_g(0) = N_g(0) exp\left(-\theta^2 / \theta_g\right),$$

where θ_g is the Gaussian parameter, defining penetration depth of positron wave functions in ion core [7, p. 337]. On this basis general curve of APAD for any material in initial approximation can be represented as:

$$N(\theta) = N_P(0)(\theta_F^2 - \theta^2) f(\theta) + + N_g(0)\exp(-\theta^2/\theta_g) + N_0 \qquad (1)$$

Normalizing constant $f(\theta)$ *in this equation has only following meanings:*

$$f(\theta) = \begin{cases} 1 & npu \quad |\theta| \le |\theta_F| \\ 0 & npu \quad |\theta| > |\theta_F| \end{cases} . \qquad (2)$$

Multiplying constants $N_p(0)$, $N_g(0)$ and N_0 in the equation (1) define respectively parabola intensity, Gaussian and level of random coincidence background. It should be pointed out that expression (1) for APAD has semiempirical nature. Area under each component (S_P, S_g) is usually defined by integration. Having value of total area under complete curve $S_0 = \int_{-\infty}^{+\infty} N(\theta)d\theta$, probability values of positron annihilation with unbound electrons and ion core electrons can be calculated respectively:

$$W_P = S_P / S_0; \quad W_G = S_g / S_0, \qquad (3)$$

as well as redistribution of probability values of positron annihilation between conduction electrons and ion excess

$$F = W_P / W_G = S_P / S_g . \qquad (4)$$

Example of decomposition of experimental spectrum on components is given in Figure 1. As parabolic component is smeared-out in domain $\theta = \theta_F$, the value of Fermi angle is usually defined by extrapolation. In all cases alterations in structure of test material are reflected in spectrum appearance and occur in the nature of stated values. For interpretation of obtained results, following values in Table 1 have been engaged: W_P, ΔW_P – probability of positron annihilation with conduction electrons and its fractional variation; θ_F - angle, corresponding with Fermi momentum; R_V – average size of structural defects; k - positron capture velocity. The latter can be estimated on the basis of positron capture model of formulas [8, p. 4634;9, p. 430; 10, p. 421]:

$$k = \sigma C_V = \frac{\bar{F} - F_f}{F_m - \bar{F}} \cdot \lambda_f \qquad (5)$$

Where σ - specific positron capture velocity; C_V - concentration of positron traps; F_f, F_m and F – annihilation values, therefore, with free positron annihilation, its maximum and current values; λ_f – free annihilation velocity.

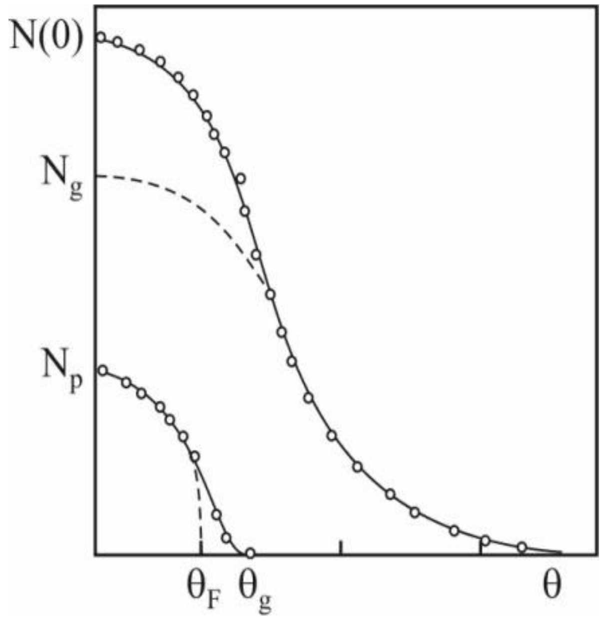

Figure 1 - Decomposition of experimental spectrum on components.

Sizes of positron traps can be defined by the expression :

$$R_V^2 = \frac{mc^2(\theta_{F0}^2 - \theta_{Fd}^2)}{8\pi\gamma}, \qquad (6)$$

where θ_{F0}, θ_{Fd} - angles, corresponding with Fermi momentum for annealed and defective material condition; γ - surface energy in defect domain (for Ti: γ = 1,39·10³ dyne/cm). Calculated in such way values k and R_V allow receiving certain presentation on qualitative change of structure resulting from irradiation and temperature-cycling (tab. 1). For convenience, the following acronyms are used in this table: ann. - annealed condition; T.C. - temperature-cycling; α 50- irradiation by α-particles E=50 MeV; P30- irradiation by protons with E = 30 MeV, and also their combinations in relevant consequence. Thermal cycling has been carried out by the following scheme: immersion of instance in liquid nitrogen with 15 min. of hold up time, and following warming in air with 2-hour hold up time.

Irradiation by charged particles results in considerable redistribution of electron density in arising defects, which is reflected in increase of W_P and decrease of θ_F. Temperature-cycling of annealed instances also results in increase of W_P and decrease of θ_F, in certain cases similar to impact from proton irradiation.

Table 1

The impact of thermal cycling on the structural characteristics of radiation-modified titanium alloys.

Composition at.%	Alloyscondition	W_P	ΔW_P, %	θ_F, mrad	R_y, A	x, $n\sec^{-1}$
Ti-1.4 In	annealing	0.171	-	6.36	-	-
	annealing +Th.c.	0.302	77	6.05	-	-
	α50	0.453	165	5.20	5.5	-
	α50 +Th.c.	0.535	213	5.57	-	25.45
	P30	0.323	89	5.75	4.15	-
	P30+Th.c.	0.390	128	5.66	-	5.25
Ti-2.9 In	annealing	0.240	-	6.44	-	-
	annealing +Th.c.	0.383	60	5.48	5.2	11.88
	α50	0.452	88	5.10	5.9	119.2
	α50 +Th.c.	0.464	93	5.20	-	-
	P30	0.314	31	5.96	3.4	3.33
	P30+Th.c.	0.427	78	5.75	-	-
Ti-5.1 In	annealing	0.243	-	6.25	-	-

	annealing +Th.c.	0.341	40	5.36	4.9	3.57
	α50	0.480	97	5.20	5.3	17.05
	α50 +Th.c.	0.566	133	5.39	-	-
	P30	0.308	27	6.00	2.7	3.37
	P30+Th.c.	0.418	72	5.48	4.6	9.67
Ti-7.4 In	annealing	0.242	-	6.04	-	-
	P30	0.322	33	5.64	3.3	1.93
	P30+Th.c.	0.462	91	5.10	4.9	12.47
Ti-10.3 In	annealing	0.272	-	6.33	-	-
	P30	0.321	18	5.92	3.4	1.24
	P30+Th.c.	0.451	66	5.75	4.0	9.51
Error ±		0.005	2	0.05	-	-

The extent of impact of thermal cycling can be estimated by value ΔF, which has regressive character from 77 to 40 % as increasing the content of In in the alloy. Our goal was to determine the reason and processes which have resulted in characteristic changes of defective condition in APAD spectrum and how its values have changed after thermal cycling. We have taken into account, that measurement of each spectrum has been carried out under ambient temperature in two hours, when all possible relaxations has likely completed after thermal cycling, and process of spectrum measurement lasts for 20 hours. Analyzing the results of thermal cycling exposure on radiated materials, it can be noted that this process has some impact on them either.

Probability of W_P annihilation increases in all cases, especially for proton radiated materials, in 2-3 times. The value of Fermi angle θ_F for α-radiated materials increases slightly, and in the second case (fig.2, curve 2) continues to decrease. On the basis of changes of annihilation values as the result of temperature-cycling, it can be considered that these changes are resulted from hardening processes of vacancy equilibrium concentration, which is available in metal under any temperature. However, this vacancy concentration under ambient temperature is certainly not sufficient for such changes in ADAP spectrum.

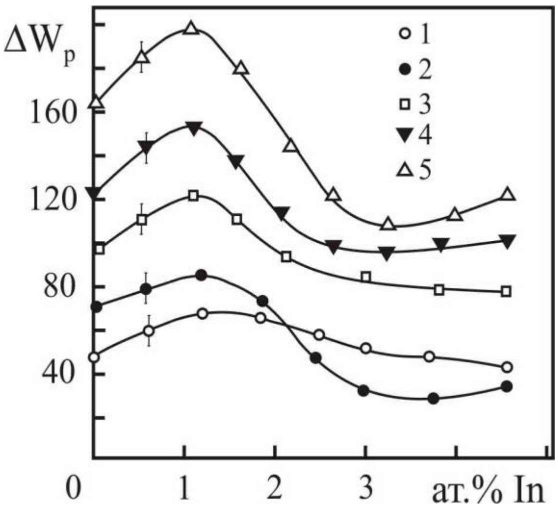

Figure 2 - The impact of radiation on the thermalcycling titanium alloys damaged from various states. 1-annealed; 2- proton radiated; 3- after protons radiated; 4-α-radiated; 5-after α-radiated.

Therefore, another more probable mechanism of observed occurrences should be offered. Under chilling and due to high thermal conductivity, metal suffers from all-around compression and, consequently, considerable subsurface stresses arises. All grains, subgrains and crystallites of metal are exposed to compression due to subsurface stresses. Besides that, dramatic negative heat stroke, which is chilling under the liquid nitrogen, can make conditions for some reorientation of separate crystallites and their grinding, as well as grain rotation. As probability of W_P annihilation for single crystals depends on shape of Fermi surface, changes in orientation of particular crystallites in polycrystalline material can result in observable changes of this value.

Due to the anisotropy of the electron momentum, such reorientation of crystallites may change and the angle of the Fermi - θ_F. Besides, various concentrations of the alloying elements in alloys and radiation defects formed as a result of irradiation α - particles, protons, and the difference in their configurations, obviously cause also various nature of grain orientation, which ultimately results in one case - to increase, and the other - to reduce the angle θ_F. It is probable that metal keeps the new internal state acquired as a result of thermal cycling, maintains both quenching and after removal of the cooling. At the same time it can be seen that the radiation defects resulting from radiation α - particles have a disordered character and have a higher capture rate of positrons than defects by proton irradiation, thus show big efficiency of positron capture as a result of thermal cycling.

Conclusions

Thus, due to differences in crystallite structure of the sample, even of the same alloy movement and rotation of grains initiated thermal shock at - apparently determined the observed irregular changes of annihilation parameters for alloys of different composition in different types of effects. As internal changes in the crystal structure revealed by thethermal cycling occurs without moving the previously created structures, these processes can be called diffused-free relaxation.

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