Copper Alloys Selected for ITER Investigated by Positron Annihilation Spectroscopy

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Abstract. The work is oriented towards the study of the high-energy neutron (proton) flux induced disorder in selected Cu-alloys by positron annihilation spectroscopy (PAS). These Cu-alloys should be applied in the reactor as a cooler and they should be used to the diffuse heat. For the simulation of the radiation damage of neutron flux, the ion implantation of protons has been applied. We supposed that the ballistic influence of protons at the primary –knocked- on atoms (PKA) production could simulate the ballistic influence of neutrons at Cu-alloys in fusion reactor ITER. Defects in the form of vacancies (loops, voids, etc.) in selected Cu-alloys were studied using pulsed low energy system (PLEPS). The selected specimens were implanted in Ion beam laboratory of FEI STU Bratislava. The energy of implantation was E_H=2x95 keV for the molecular H_2^+ ion beam. Two implantation doses were chosen for both of the alloys: 1.3x10^{19} ions/cm^2 (1.1 C/cm^2) and 5x10^{18} ions/cm^2 (0.4 C/cm^2). Using PLEPS a depth profiling and a void creation (probably filled with H_2) in the area from 50-480 nm was observed. Although the influence of neutrons with energy 14 MeV and protons with energy 95 keV is not the same (differences in energy and existence of proton charge), the experimental simulation (for the range where protons and neutron are not thermalised) of radiation damage of ITER construction materials was successfully performed. After isochronal annealing of both materials in vacuum in range 100-600ºC, the recovering of defects in CuCrZr was much more effective than in CuAl25.

1. Introduction

The particle bombardment of copper alloys induces various changes in mechanical properties. Therefore, investigation of created defects is an interesting issue for solid-state science and applied research. The problem of defect accumulation in Cu-alloys is interesting in different areas of industry (electronics, nuclear fusion technology, etc.). For the optimal application of these materials it is necessary to know a changes in microstructure due to treatment via different external factors (temperature changes, ageing, particle irradiation, etc.).

Development of nuclear fusion as a practical energy source could provide great benefits. This fact has been widely recognized and an investigation of nuclear fusion materials has enjoyed a high level of international co-operation.

For our investigation two families of copper alloys were selected. They are considered for the first wall materials for the International Thermonuclear Experimental Reactor (ITER) and both exhibit high thermal conductivity and strength and radiation resistance [1].

The precipitation hardened (PH) alloys (for example CuCrZr) represent a wide family of heat-treatable alloys. PH alloys reach an optimum strength after the thermomechanical treatment involving a solution annealing at high temperature to dissolve the alloying elements, then a water quench to keep the alloying elements in supersaturated solid solution at room temperature and finally an ageing treatment at intermediate temperatures to decompose the supersaturated solid solution.

The dispersion strengthened (DS) copper alloys (for example CuAl25) are powder metallurgy products, characterised by a fine dispersion of nanometric second phase particle that give strength and thermal stability to the copper matrix. Their properties strongly depend on dispersed phase type, dimension and volume fraction, production method, and degree of cold working, and are less sensitive to subsequent heat treatments [1]. Our main work is oriented towards the study of the high-energy neutron (proton) flux induced disorder in Cu-alloys by positron annihilation spectroscopy. These Cu-alloys should be applied in the reactor as a cooler and they should be used to the diffuse heat.

For the simulation of the radiation damage of neutron flux, the ion implantation of protons has been applied. The protons were chosen having approximately the same mass as that of a neutron. We supposed that the ballistic influence of protons at the primary-knocked-on atoms (PKA) production could simulate the ballistic influence of neutrons at Cu-alloys in fusion reactor ITER.

Our group performed an atomic simulation of grain boundary sliding and migration in copper, too. In this study, we present a Monte Carlo investigation, based on the Embedded Atom Method potential, of the $\Sigma5$ grain boundary sliding in copper at elevated temperature. Important aspect of this approach is the implementation of simulated annealing technique. We find a variety of sliding behaviours, including coupling to migration. While our previous results showed that elevated temperature in aluminium $\Sigma5$ GB increases the rate of migration, we did not find a temperature dependency of interface migration in copper. Detailed informations about this topic were already published in [3].

Defects in the form of vacancies (loops, voids, etc.) in selected Cu-alloys were studied using Positron Annihilation Spectroscopy (PAS). A pulsed low energy system, developed at the Institut für Nukleare Festkörperphysik, Universität der Bundeswehr, Neubiberg/München was used [2].

The positron lifetime technique is a well-established method for studying open-volume type atomic defects and defect’s impurity interactions in metals. The lifetime of positrons trapped at radiation-induced vacancies, vacancy-impurity pairs, dislocations, microvoids, etc. is longer than that of free positrons in perfect region of the same material. As a result of the presence of open-volume defects, the average positron lifetime observed in structural materials is found to increase with the damage [4,5].

Cu-based alloys are supposed to serve as a basis to construct heat sinks for first wall and divertor components of ITER. CuCrZr and CuAl25 alloys are most promising among such materials. To our knowledge, this is the first positron annihilation study of irradiated CuCrZr and CuAl25 materials. PAS techniques are especially sensitive to open volume defects like vacancies and their clusters. In solids positrons reside mainly in the interstitial space because the repulsion of nuclei is weak here. In open volume defects this repulsion is even weaker and positrons may get trapped is such defects because they constitute a potential well for positrons. Irradiation damage is usual way of production of vacancies and their agglomerates that are typical examples of open volume defects.

PAS techniques can also be used to detect and investigate clusters embedded in a matrix. The necessary condition for positron trapping is that such clusters represent a potential well for positrons. Positron trapping at precipitates and other embedded particles may be of importance in CuCrZr and CuAl25 materials. These systems exhibit complicated microstructure with various particles surrounded by the Cu matrix.

2. Experimental

The improved pulsed low-energy positron system (PLEPS) seems to be the optimal technique for the evaluation of defects structure and concentration in Cu-alloys. This system enables to study the micro structural changes in the region from 20 to 500 nm (depth profiling) with relatively small specimens of about 8x8x0.5 mm^3. The resolution of PLEPS was about 240 ps FWHM [2,6]. All spectra contained about $3x10^7$ events at a peak to background ratio of about 5000:1 [2]. The accuracy of the lifetime values is of about ±1 ps. Before an implantation, all specimens CuCrZr and CuAl25 were polished to the "mirror-like" state (grain size < 1 μm) and measured using PLEPS in vacuum better than 10^{-6} Pa. In total, 6 specimens were studied (see TABLE I).

The selected specimens were implanted in Ion beam laboratory of FEI STU Bratislava [7]. The energy of implantation was E_H=2x95 keV for the molecular H_2^+ ion beam. Two implantation doses were chosen for both of the alloys: $1.3x10^{19}$ ions/cm^2 (1.1 C/cm^2) and $5x10^{18}$ ions/cm^2 (0.4 C/cm^2). For these doses the surface sputtering could be neglected as it is about 2 and 1 mono-layers, respectively (calculated sputtering yield for 95 keV protons in Cu-alloys is $Y=1x10^{-3}$ at/ion). For the protons with the energy of 95 keV, the range of 480 nm was found by TRIM calculations. This range was chosen with regard to the scope of the PLEPS equipment. The PAS-PLEPS spectra were evaluated using program POSWIN [2].

TAB. I: SPECIMENS CHEMICAL COMPOSITION AND IMPLANTATION DOSE.

Specimen	**Material**	**Implantation dose [C/cm^2]**
SS	CuCrZr – 98.95w.%Cu; 0.6-0.9w.%Cr; 0.07-0.15w.%Zr	None
SA	CuCrZr	0.4
SM	CuCrZr	1.1
TT	CuAl25 – 99.5w.%Cu; 0.25w.%Al as Al_2O_3; 0.22w.%O as Al_2O_3; 0.025w.%B as B_2O_3	None
TA	CuAl25	0.4
TM	CuAl25	1.1

3. Results and conclusion

Results are summarised in *Fig.1* and *Fig.2.* The differences between both materials are clearly observable. The main parameter – positron mean lifetime (MLT) can be split in to two components (τ_1 and τ_2 with intensities I_1+I_2=100%) in all experimental spectra. τ_1 indicated changes in the bulk with the simple defects like mono- or di-vacancies, τ_2 gives information about large vacancy clusters or voids in the material. From the PAS analyse point of view, the CuAl25 material with MTL of about 230 ps and intensity of τ_2 component on the level of 13% in the non-implanted stage contains much more vacancy agglomerations than non-implanted CuCrZr reference material with MLT of about 175 ps and intensity of τ_2 on the level of 3%.

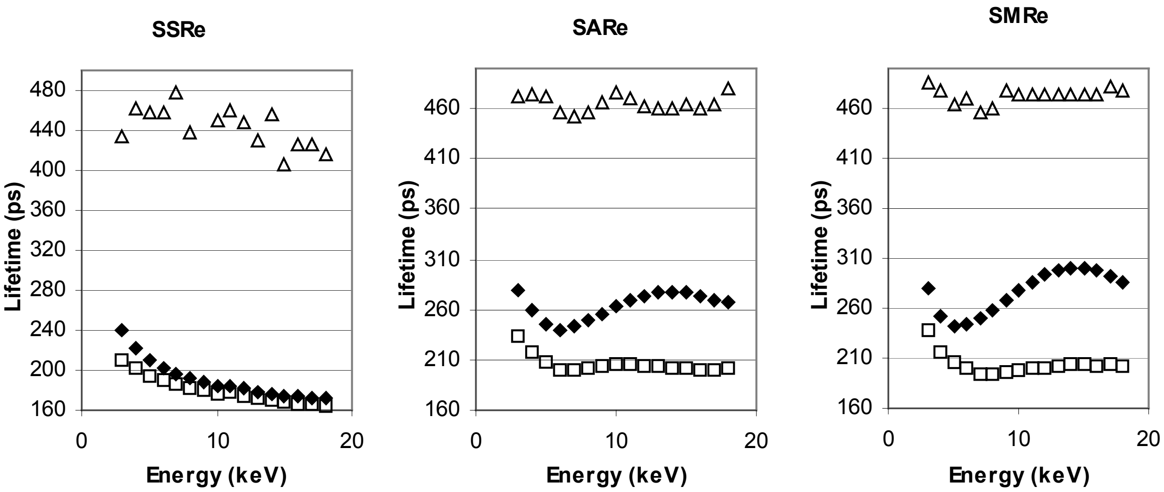

Fig. 1 – Comparison of positron lifetimes in CuCrZr samples implanted by protons. SSRe - not implanted, SARe - implanted to the dose 0.4C/cm^2, SMRe – implanted to the dose 1.1C/cm^2. Symbols: ♦- Mean lifetime, □- τ_1, Δ- τ_2.

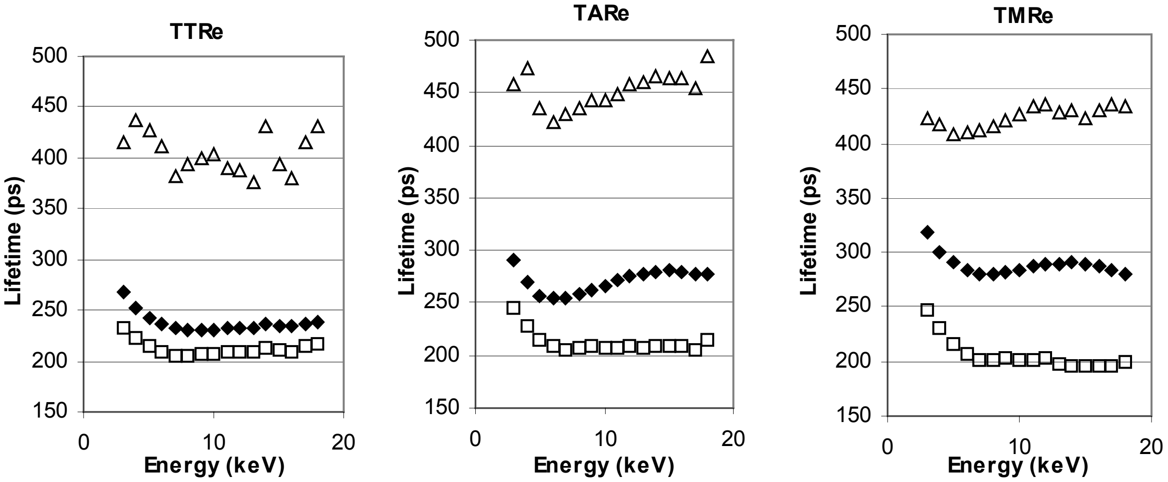

Fig. 2 – Comparison of positron lifetimes in CuAl25 samples implanted by protons. TTRe - not implanted, TARe - implanted to the dose 0.4C/cm^2, TMRe – implanted to the dose 1.1C/cm^2. Symbols: ♦- Mean lifetime, □- τ_1, Δ- τ_2.

Due to the implantation dose an increase of MLT was identified in all implanted specimen spectra. The maxima of MLT at all implanted samples (independent on implantation dose) are at positron energy of about 13 keV. According to the equation

$$z_0(E) \approx \frac{40}{\rho}[E / keV]^{1,6}$$

this energy can be assigned to the distance from the surface $z_o(E)$ of about 270 nm. That means, after implantation procedure, a creation of large vacancy agglomerations (voids) was observed. The maximal values of I_2 are on the level of 35% (CuCrZr) and 40% (CuAl25), respectively. According to the fact, that the changes in τ_1 values are at all specimens after implantation minimal, it can be concluded, that:

- Using PLEPS a depth profiling and a void creation (probably filled with H_2) in the area from 50-480 nm was observed.

- Although the influence of neutrons with energy 14 MeV and protons with energy 95 keV is not the same (differences in energy and existence of proton charge), the experimental simulation (for the range where protons and neutron are not thermalised) of radiation damage of ITER construction materials was successfully performed.

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