NON-DESTRUCTIVE CHARACTERIZATION OF THE MATERIALS FOR FUTURE NUCLEAR REACTORS

***J. Snopek*[1]*, V. Slugeň*[1]**

[1]*Institute of Nuclear and Physical Engineering,*
Faculty of Electrical Engineering and Information Technology,
Slovak University of Technology in Bratislava,
Ilkovičova 3, 812 19 Bratislava, Slovak Republic
(E-mail: jozef.snopek@stuba.sk, vladimir.slugen@stuba.sk)

Received 03 May 2016; accepted 12 May 2016

1. Introduction

Today's reactor technology is being criticized for low efficiency and amount of radioactive waste they produce. Today's most modern reactors (3rd generation) have improved safety over reactors of last generation however they do not improve much in mentioned fields. There is therefore a need for the new generation of reactors (4th generation), which could address these challenges. When material is heated its lattice accepts energy. Heat may cause some element to precipitate in steel or just introduce vacancy defects and/or dislocations. It also may anneal the lattice, which depends on temperature. If heat knocks out an atom out of its original position in material lattice that could cause internal stresses of material and eventually change in macroscopic characteristics, like durability.

For our experiments, we have used Barkhausen noise technique, which is powerful non-destructive method for monitoring stresses in lattices of magnetic materials. We have also used PAS, which is powerful non-destructive method for diagnosing vacancy defects in variable materials. We researched some ODS steels, which are primarily going to be used as fuel cladding or reactor pressure vessel internal components.

1.1. ODS steels

Oxide dispersion strengthened steels (ODS) are progressive materials manufactured by mechanical alloying [1]. The radiation resistance and creep resistance at high temperatures is ensured by thermally stable oxide particles dispersed in ferritic matrix. The ODS steels are unique for their nano-size oxide particles (Y-Ti-O atoms) and for controlled micron-size grain morphology. These small nanocluster particles not only hinder dislocation motion, but they are also effective sinks for radiation-induced defects [2]. ODS steels have been developed for high temperature applications like heat exchangers in conventional power plants. In addition to superior creep properties at temperatures up to 1200 °C these materials generally show excellent corrosion and swelling resistance when irradiated by neutrons [3].

1.2. Testing techniques

1.2.1. Barkhausen Noise

Magnetic Barkhausen Noise (BN) analysis is based on a concept of inductive measurement of a noise-like signal, generated when magnetic field is applied to a ferromagnetic sample. BN is sensitive to various parameters which affect the magnetic domains configuration and domain-wall pinning sites. These parameters are particularly grain size, different phase precipitates, surface conditions, hardness, residual stress and fatigue but also by magnetic field strength and applied stress [4, 5]. In addition to the industrial application (aviation, pipeline transport, automobile industry) [6, 7], this technique can be well used for the material performance assessment of the nuclear installations [8].

1.2.2. Positron annihilation spectroscopy

Positron Annihilation Spectroscopy is a powerful tool for investigation of materials microstructure. By this method it is possible to predict material structure defects with the size of 0.1 to 1.0 nm provided defects are connected with less than average density of electrons, for example dislocations, dislocation loops, vacancies and clusters of vacancies. In principle this method is able to provide useful information even about defects like precipitation with higher (more negative) positron affinity (A+) than the bulk material, e.g. for ODS steels, yttrium (positron affinity A+ = -5.31) oxides are being used as obstacles for dislocation motion in Fe-Cr matrix (A+ of Fe and Cr =-3.84 and -2.62 respectively [9].

2. Outline of material challenges

The objects of our study were various oxide-dispersion strengthened (ODS) steels, namely PM2000, MA956, MA957 and ODM751 as well as ODS variant of Eurofer steel. Chemical compositions of the studied materials are listed in Table 1 (appendix). These materials are strong candidates for structural materials used in Generation IV reactors which have to endure extreme service conditions. Service conditions that characterize these reactors include:

1. Operating temperature above 700 °C
2. High neutron doses
3. Highly corrosive environment

Requirements for materials for GEN IV reactors are:

1. Structural stability
2. High radiation (Small defects, good recombination), thermal (high, melting temperature, stability at high temperature, low coefficient of thermal length and volume expansion) and corrosion resistance (surface passivation).
3. High resistance to cyclic strain (thermal, mechanical)
4. High thermal conductivity
5. Reduced activation (low content of chemical elements with long half lifetime after neutron capture, low cross section of neutron absorption)
6. Improved mechanical properties and resistance to thermal shock (high strength, high toughness, enough hardness, low brittleness, low material abrasion and erosion)
7. Long lifetime and low ageing [10].

In order to induce the intermediate temperature embrittlement, all recrystallized samples as well as ODS Eurofer have undergone isothermal annealing treatment at 475 °C for 100, 500 and 1000 h in high temperature furnace, LECO HT 1600, under Ar atmosphere of 5 mBar overpressure. At this temperature a phase separation of ferrite phase into Fe-rich α and Cr-rich α’ phase occurs. The reason of this so called 475 °C embrittlement phenomenon is the miscibility gap in the Fe-Cr equilibrium system [11].

In order to distinguish the effect of α' precipitation from other thermal annealing effects, analogical thermal aging at 650 °C was performed. Phase separation at this temperature does not take place and material generally does not suffer from embrittlement.

3. Main results

3.1.1. Barkhausen Noise results

Although the industrial application of magnetic Barkhausen noise technique is usually based on changes in the RMS parameter, in a complex material characterization based on the BN technique it is necessary to consider also the qualitative parameters of the signal, such as peak position and FWHM as well as frequency spectra of the noise. As it was outlined in the previous chapters, these parameters might be used as an indicator of concentration and distribution of microstructural defects. In order to verify these assumptions and contribute to the characterization of ODS materials, comparative measurements of various ODS materials were performed.

PM2000, MA956, ODM751, ODS EUROFER as well as conventional EUROFER97 were investigated in form of 10x10x0.5 mm samples. In the case of MA957 material, 8x8x0.5 mm sample was used. Based on the previous experiments, we used 100 Hz magnetizing field for the comparative BN characterization of different ODS materials. In all measurements discussed in this chapter external magnetic field was oriented perpendicular to the grain elongation of the materials. Detailed experimental parameters settings are listed in the Table 2 (appendix). The averaged results of the BN signal parameters from at least five measurements are listed in the Table 3 (appendix). The averaged signal envelopes are shown in the Fig. 1

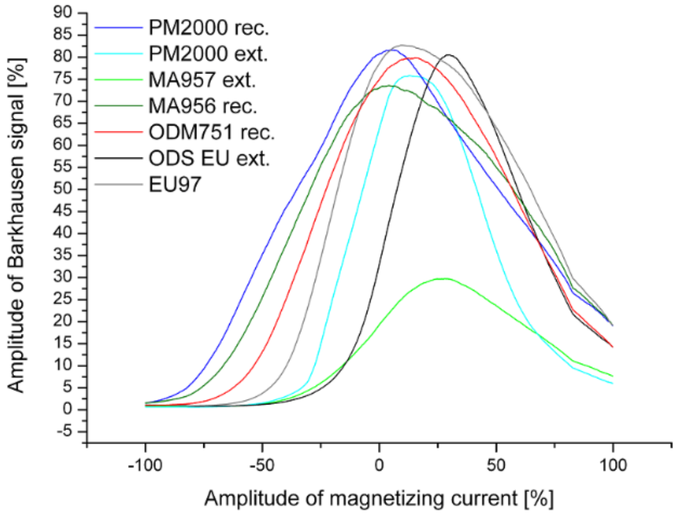

Fig. 1 BN noise signal envelopes measured for different ODS materials and Eurofer 97.

As can be seen in the Fig. 1, there is no clear difference in peak height and RMS values between as-extruded and recrystallized materials. Lower BN signal from the MA957 material can be attributed to the size of the sample, which was 8x8x0.5 mm (against 10x10x0.5 mm in all other materials). On the other hand, the FWHM and peak position seem to be dependent on the concentration of microstructural imperfections given by chemical composition and temperature history.

As can be seen in the Table 3 (appendix), materials studied in our experiments exhibit different values of FWHM ranging from 52 to 127 % of half-period of magnetizing field. In addition to this, peaks of the BN signal with higher FWHM (recrystallized materials) are shifted to the smaller values and vice versa. This indicates the recrystallized materials contain a larger spectrum of weaker obstacles, while as-extruded materials to contain smaller spectrum of rather strong obstacles. As one of the strong non-magnetic obstacles for domain wall movement, considered in the studied materials, is yttrium (yttrium aluminium) oxide, we assume that its characteristics determine the shift and width of BN signal. Consequently we can conclude that a rather wide range of coarsened oxide particles in the recrystallized materials act as weaker obstacles for magnetic domain wall movement and results in wider BN signal with relatively small shift of the peak. On the other hand, high concentration of fine oxide particles, characterizing as-extruded ODS materials, pin the domain walls stronger and the result BN signal is narrower and shifted to the higher values of magnetizing field.

Although the oxide particles in the studied materials seem to be important pinning sites for domain wall movement, their diversity does not affect BN frequency spectrum significantly Fig. 2. shows frequency spectra of all investigated materials. All materials are characterized with BN noise frequency 50 – 200 kHz. Although the amplitudes of noise spectra differ between materials (Fig. 2a), the course of the frequency spectra is similar for all materials (Fig. 2b).

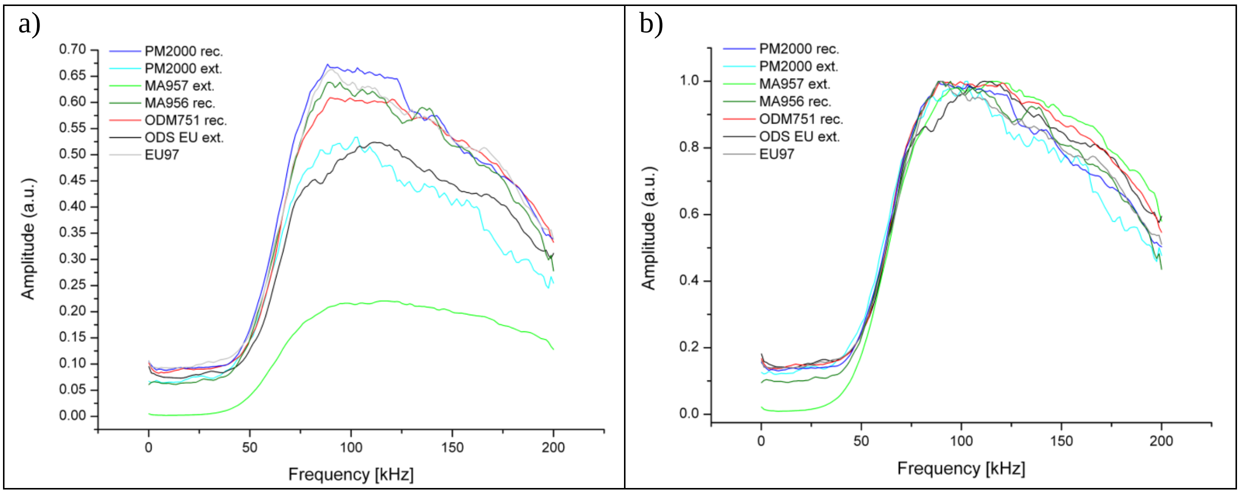

Fig. 2 BN noise frequency spectra of the measured materials smoothened (a.) and smoothened + normalized (b.).

Series of Barkhausen noise measurements on the materials aged at 475 °C were conducted. In order to distinguish in the measured parameters between the effects of precipitation of α’ and the thermal annealing of the lattice, analogical thermal aging at 650 °C was performed. Based on the previous obtained results and assuming the α’ precipitates to be pinning sites for magnetic domain walls movement, the position of the BN peak for different aging times of recrystallized PM2000 and MA956 materials (Fig. 3) have been investigated. The experimental results in the form of individual BN signal parameters are listed in the Table 4 (appendix).

As can be seen in the Fig. 3, both materials show different behavior for 475 °C and 650 °C aging. While the 650 °C thermal treatment leads to only relatively small increase of BN signal shift in PM2000 and significant decrease in MA956, 475 °C aging significantly increases the shift of both materials.

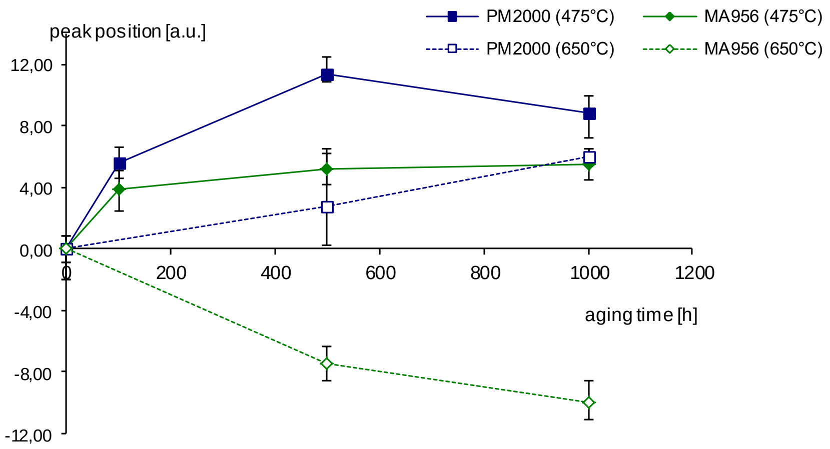

Fig. 3 Position of the BN peak obtained from heat treated MA956 material.

We assume that the shift of the BN signal to higher magnetizing field values is due to precipitation of new phase, which acts as stronger obstacle for domain wall movement. This assumption is in agreement with Moorthy et al. who observed second phase precipitates in Cr-Mo ferritic steel at higher field strength only [12].

3.1.2. Positron annihilation spectroscopy

In this thesis MA956 and PM2000 were primarily investigated by three-component decomposition of the spectra. They were aged by 475 °C and 650 °C. Sample of EU97 (aged at 475 °C for 100 h) was measured for comparison to ODS Eurofer. The lifetime spectra were fitted with a variance of fit (FV) ranging from 0.9961 to 1.0934 after source correction. As can be seen in Table 5(appendix), no presence of dislocations (or very small dislocation density respectively) has been observed in these materials and the first lifetime component is mostly reduced or fixed to accomplish better variance of fit.

As it may be seen in Table 5(appendix), Fine grained sample of PM2000 has a defect component (LT2) smaller, than coarse grained sample of PM2000. Positron lifetime around ~ 198 ps corresponds to both small vacancy defects (mono and di-vacancies) and annihilation in grain boundaries. This is also supported by relatively high intensity of 78.2 %. As for coarse grained samples a longer component of ~240 ps (and 224 ps in MA956) was found. Its value can be assigned to both yttria nanoparticles [13] and small clusters with the mean size of 3-5 vacancies which are a result of mechanical alloying procedure [14, 15].

As can be seen from MLT it is clear that recrystallized PM2000 is the material with the lowest concentration of defects despite having the biggest defects in size and PM2000 Fine grained is the material with the highest concentration of defects of the three. However, these defects (grain boundaries) are the basic structure of this material. Also even though defects are very small in this sample, intensity of defects over 78 % makes it the material with the highest MLT. In Fig. 4 first two components along with MLT are displayed.

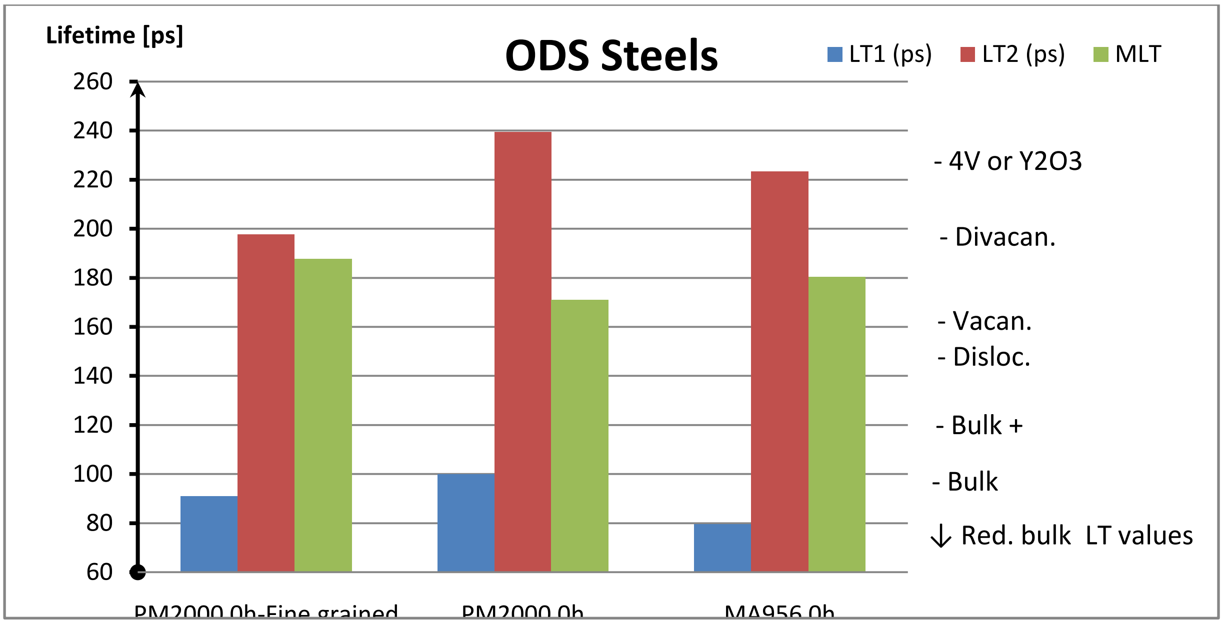

Fig. 4 Positron lifetimes and MLT in the investigated materials.

The ODS steels samples were also aged at 475 °C for 100 and 500 hours and 650 °C for 500 hours. Sample of Eurofer-97 aged at 475 °C for 100 h was compared to ODS steels. Measured results are in Table 6 (appendix).

Measurements of positron lifetimes in the ODS materials aged at 475 °C show the evolution of vacancy type defects with annealing time. Introducing thermal aging for 100 hours to these samples actually had a positive effect. Intensity of defects has been reduced in both PM2000 and MA956 compared to original materials and for PM2000 size of vacancy type defects has been reduced as well, since the coarsening of oxide particles below ~1100 °C is negligible [16]. However reduced bulk time for MA956 has slightly risen which may point out creation of small dislocations. MLT of aged MA956 doesn't show much difference compared to original sample, however MLT of PM2000 has decreased, which can be attributed to recombination of atoms back into 4V defects. Compared to EUROFER97 which turned out to have smaller defects (size of mono and di-vacancies) with about half of positrons annihilating in them and also lower MLT

Aging at 475 °C for 500h has shown an overall increase in MLT. This can be associated with higher fraction of positron trapping at vacancy clusters and also increase in LT_2 (~240 ps) is associated with yttrium nanoparticles and small vacancy clusters with the mean size 4-6 vacancies [16]. Results are in Table 7 (appendix). Although lifetimes for defects have rapidly risen in MA965, intensity decreased which could be the sign of annealing of defects which move in the lattice or are grouping together from smaller sized defects to larger ones. Diffusion process is aided with elevated temperature.

To see the effects of higher temperature to these ODS materials similar aging to 650 °C for 500 hours was carried out. Results are presented in Table 8 (appendix).

Imperfection and small defects that contributed to bulk lifetime value of around 100 ps has distinguish themselves after aging at 650 °C in PM2000 and contributed to LT_2 instead, probably with occurrence of new thermo-vacations. Therefore intensity of annihilation in bulk has decreased to ~ 28% and reduced bulk lifetime is only 67 ps. Higher concentration of defects with lifetime of around 220 ps is considered to be 3-vacancies and 4-vacancies. Bulk parameter for MA956 was fixed for better spectra decomposition and time of 243 ps is attributed to 4-vacancies and yttrium nanoparticles. MLT has raised significantly in PM2000 and only slightly in MA956 over 475 °C aged samples.

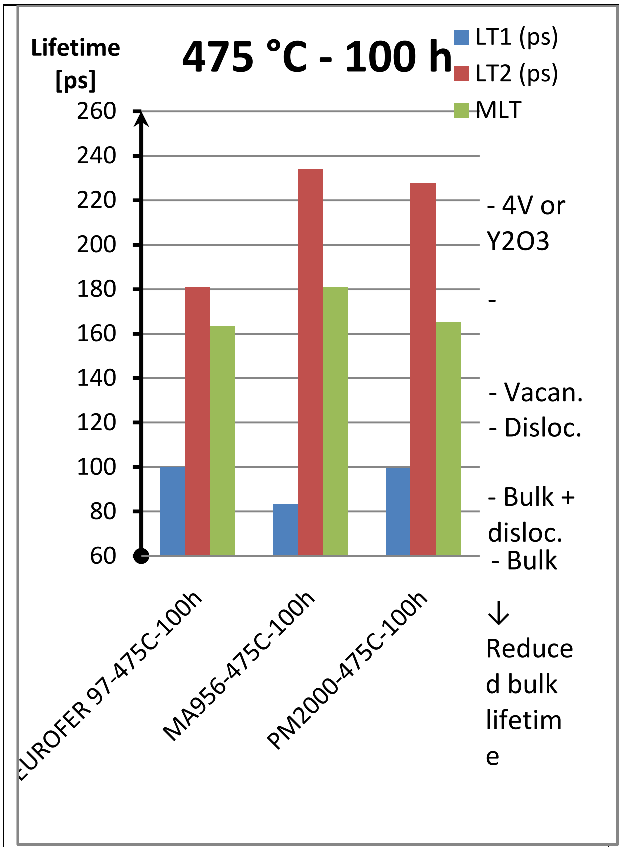

Fig. 5 Positron lifetimes and MLT in the investigated materials aged at 475 °C for 100h.

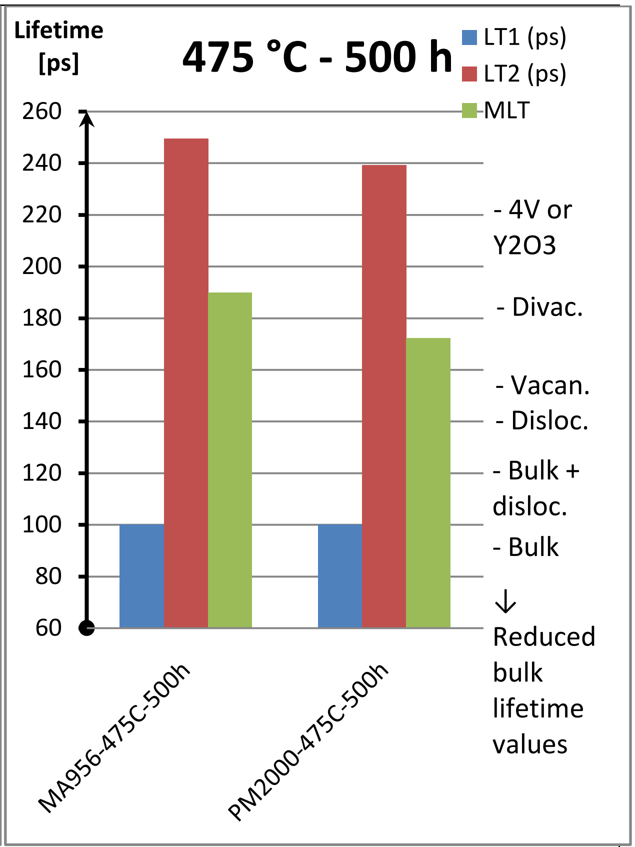

Fig. 6 Positron lifetimes and MLT in the investigated materials aged at 475 °C for 500 hours.

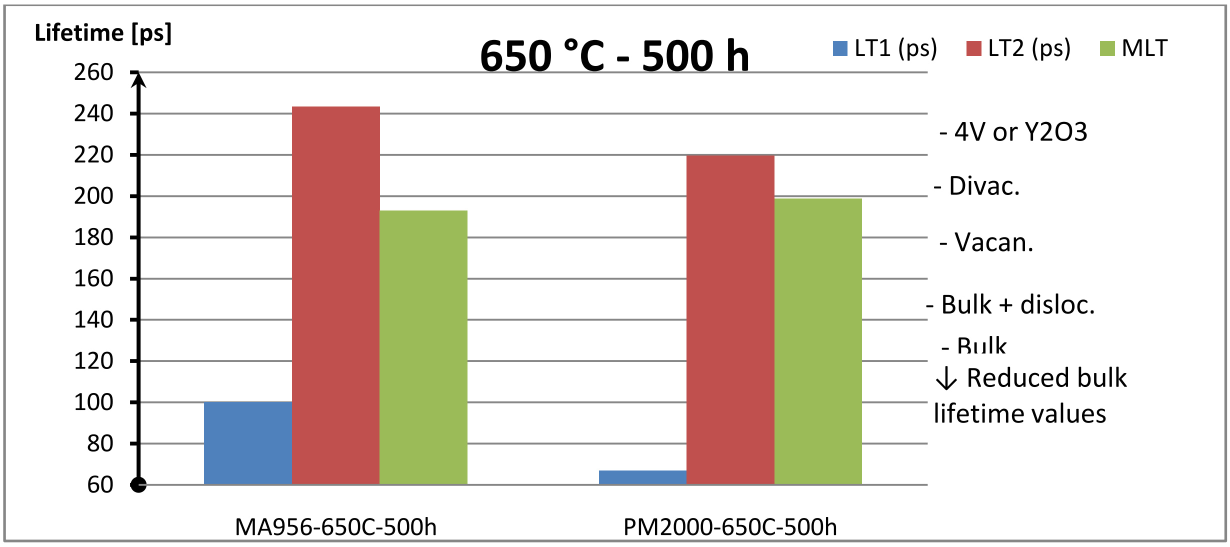

Fig. 7 Positron lifetimes and MLT in the investigated materials aged at 650 °C for 500 hours.

4. Example

In the paper by [12] authors discuss two groups of defects in the ER36 gear steel observed in Barkhausen noise measurements. First group of defects – weaker obstacles can be well investigated with a wide frequency spectrum of magnetizing field (4 – 125 Hz). For instance, boundary of ferrite grain belongs here. Second group of defects, like carbides are considered as stronger obstacles and they can be observed only in the BN spectra induced by lower frequencies of magnetizing field. It is assumed that lower frequencies lead to less significant eddy current opposition and therefore to a stronger magnetization. Experiments of Moorthy et al. with sufficiently strong effective magnetizing field (induced by frequency of 4 Hz) result in an additional peak in BN spectrum, which is shifted to higher magnetizing field values. In other words we can say that the resulting BN spectrum, characterized by more than one peak is induced by more than one kind of domain wall obstacles or two types of iron phases. Consequently we can conclude that a larger spectrum of obstacles/defects leads to a wider BN signal, characterized by higher FWHM values.

5. Conclusion

This thesis describes the behavior of the microstructure of the oxide dispersion strengthened steels at intermediate temperature. Two, in principle, different techniques were used for the characterization of the microstructure of the oxide dispersion strengthened steels thermally aged at 475 °C and 650 °C. Both techniques, namely Positron annihilation lifetime spectroscopy (PAS) and Barkhausen noise (BN) measurements are very sensitive to metallurgical modifications and presence of nano-sized imperfections in the crystal lattice. Precipitation of the nano-sized α' phases shift the Barkhausen noise signal. The main conclusions obtained from the experiments are summarized below:

- Although the positron lifetime technique is not directly sensitive to the Cr- rich α' phase in the Fe lattice, the experiments showed that these precipitates affect the mobility of point defects and consequently change the annihilation characteristics.

- It is reasonable to assess the metallurgical changes of the various materials through the FWHM and BN peak shift values. In general, stronger obstacles for domain wall movement shift the BN peak to higher values of magnetizing field while larger spectrum of defects results in a wider BN signal (higher FWHM value).

- Higher concentration of finer oxide particles, typical for as-extruded ODS materials shift the BN signal to higher values of the magnetizing field. This indicates that these defects are stronger obstacles for domain wall movement than the coarsened particles (with correspondingly lower concentration), typical for recrystallized ODS materials.

- Thermal aging of the ODS materials at 475 °C leads to a new type of strong obstacles, shifting the BN signal to higher values of magnetizing field. Thermal aging at 650 °C increase the peak shift only little (PM2000) or decrease the value respectively (MA956).

Bibliography

[1] C. Suryanarayana, "Mechanical alloying and milling," *Progress in Materials Science,* vol. 46, no. 1-2, pp. 1-184, 2001.

[2] V. Sabelova, Study of the radiation resistance of alloys based on De-Cr, Bratislava: FEI, 2014.

[3] R. L. Klueh and D. R. Harries, High-Chromium Ferritic and Martensitic Steels for Nuclear Applications, ASTM, 2001.

[4] J. Degmová and L. Debarberis, "Magnetic Barkhausen noise measurement of ascast model steels with parametric variation of Ni, Mn and Si content," Directorate general: Joint Research Centre, Petten, 2006.

[5] J. VETERNÍKOVÁ, J. DEGMOVÁ, S. KILPELÄINEN, V. Slugeň, F. Tuomisto, J. Räisänen, V. Kršjak, M. Petriska, S. Sojak, R. Hinca and M. Stacho, "Non-destructive examination of helium implanted HTRs construction materials," *Nuclear Engineering and Design,* vol. 251, pp. 354-359, 2011.

[6] J. T. EVANS, B. SHAW, L. SUOMINEN and A. WOJTAS, "Detection of Thermal Damage IN Steel Components After Grinding Using the Magnetic Barkhausen Noise Method," in *ECNDT '98 - PROCEEDINGS online,* COPENHAGEN, 1998.

[7] M. WILLCOX and T. MYSAK, "An Introduction to Barkhausen Noise and its Applications," Insight NDT Equipment LTD, Herefordshire, 2000.

[8] D. PARK, S. PARK, J. JU and et. al., "Dependence of Barkhausen noise in the neutron irradiated reactor pressure vessel steel," *J. Magn. Magn. Mater,* Vols. 272-276, no. 2, pp. 272-276, 2004.

[9] M. PUSKA, P. LANKI and R. NIEMINEN, "Positron affinities for elemental metals," *Journal of Physics: Condensed Matter,* vol. 1, no. 35, pp. 6081-6093, 1989.

[10] J. Šimeg-Veterníková, "Material issues for Gen-4 reactors - R&D synergy with fusion," in *French Slovak Winter Seminar "Generation-IV Nuclear Reactors",* Bratislava, 2014.

[11] P. J. Grobner, "The 885° f (475° c) embrittlement of ferritic stainless steels," Springer, New York, 1973.

[12] V. MOORTHY, B. SHAW and J. EVANS, "Evaluation of tempering induced changes in the hardness profile of case-carburised EN36 steel using magnetic Barkhausen noise analysis," *Ndt & E International,* vol. 36, no. 1, pp. 43-49, 2003.

[13] L. C. Damonte, M. A. Taylor, J. Desimoni and J. Runco, "PALS study on the defect structure of yttria-stabilized zirconia," *Radiation Physics and Chemistry,* vol. 76, no. 2, pp. 248-251, 2006.

[14] M. J. Alinger, S. C. Glade, B. D. Wirth, G. R. Odette, T. Toyama, Y. Nagai and M. Hasegawa, "Positron annihilation characterization of nanostructured ferritic alloys," *Materials Science and Engineering A,* vol. 518, no. 1, pp. 150-157, 2009.

[15] J. SCHNEIBEL, Z. LU and S. SHIM, "Nanoprecipitates in Steels," in *BREAKTHROUGH CONCEPTS,* Oak Ridge, 2007.

[16] V. KRŠJAK, Z. SZARAZ and P. HÄHNER, "Positron annihilation lifetime study of oxide dispersion strengthened steels," *Journal of Nuclear Materials*, vol. 428, no. 1-3, pp. 160-164, 2012.

[17] H. K. D. H. Bhadeshia, "Recrystallisation of practical mechanically alloyed iron-base and nickel-base superalloys," *Materials Science and Engineering: A*, vol. 223, no. 1-2, pp. 64-77, 1997.

Appendix

Table 1 Nominal chemical compositions [wt%] of the studied materials*[17]*

Material	Form	Manufacturer	Cr	Al	Ti	Mo	W	Mn	V	Y_2O_3
PM2000	1, 2	Plansee	20	5.5	0.5	-	-	-	-	0.5
MA956	2	INCO	20	4.5	0.5	-	-	-	-	0.5
ODM751	2	Dour Metal	16.5	4.5	0.6	1.5	-	-	-	0.5
MA957	1	INCO	14	-	0.9	0.3	-	-	-	0.25
(ODS) Eurofer	1	Plansee	8.9	-	-	-	1.1	0.4	0.2	(0.3)

Form: 1 - as extruded; 2 –recrystallized

Table 2 Set parameters for BN measurements.

Frequency [Hz]	100
Magnetizing voltage [V]	3
Number of bursts	6
Sampling frequency [MHz]	5
Band-pass filter [kHz]	0 – 200

Table 3 BN signal parameters of the measured materials.

	RMS	σ	Peak	σ	Peak position	σ	FWHM	σ
PM2000 rec.	57.65	3.94	78.45	3.30	4.43	0.70	127.05	25.34
MA956 rec.	51.60	0.47	73.54	0.54	6.57	0.87	112.38	1.18
ODM 751 rec.	51.76	0.95	79.79	2.30	14.62	0.77	94.38	1.50
PM2000 extr.	41.77	0.37	76.46	1.11	16.48	0.20	61.53	1.09
MA957 extr.	18.10	3.16	29.65	6.90	27.00	2.53	80.32	11.01
ODS EUROFER	43.12	0.57	79.46	1.40	31.76	0.21	52.92	15.71
EUROFER 97*	53.79	0.50	83.60	1.06	15.50	0.86	92.87	1.31

* Conventional Eurofer 97 was measured as a reference material

Table 4 BN signal parameters of the thermally aged materials.

	Temperature [°C]	Time [h]	RMS	σ	Peak	σ	Peak position	σ	FWHM	σ
PM2000	As-received	-	57.65	3.94	78.45	3.30	4.43	0.70	127.05	25.34
	475	100	47.88	0.28	60.16	0.46	10.03	1.42	139.95	1.44
		500	43.52	0.30	59.63	0.52	15.84	0.89	119.59	1.32
		1000	52.78	0.14	69.81	0.36	13.32	1.47	129.57	0.27
	650	500	52.55	0.37	75.85	1.46	7.24	2.76	116.83	6.06
		1000	51.98	0.10	76.47	0.06	10.47	0.44	112.20	0.67
MA956	As-received	-	51.60	0.47	73.54	0.54	6.57	0.87	112.38	1.18
	475	100	49.33	0.53	70.99	1.16	10.43	1.35	110.20	0.95
		500	52.41	0.41	76.18	0.49	11.81	1.17	108.84	0.99
		1000	57.49	0.26	79.16	0.78	12.11	1.01	118.55	0.76
	650	500	54.83	0.20	76.62	1.21	-0.94	0.78	116.79	2.01
		1000	55.44	0.34	77.73	1.10	-3.49	1.03	117.40	1.07

Table 5 PALS results, MLT is the positron mean lifetime given as weighted average of three lifetime values. Annihilation in the air is not present in this table.

Sample	I_1 (%)	I_2 (%)	ΔI_1 (%)	ΔI_2 (%)	LT_1 (ps)	LT_2 (ps)	ΔLT_1 (ps)	ΔLT_2 (ps)	MLT	FIT
PM2000 0h-Fine grained	19	78.2	1.7	1.8	91	197.7	1.7	4.2	187.796	0.9961
PM2000 0h	53.83	45.54	0.54	0.54	100	239.5	0	3.3	170.9623	0.9971
MA956 0h	38.2	59.5	1.6	1.6	79.6	223.4	4	5.8	180.5281	1.0429

Table 6 PALS results of 475 °C aged samples for 100 h, MLT is the positron mean lifetime given as weighted average of three lifetime values. Annihilation in the air is not present in this table.

Sample	I_1 (%)	I_2 (%)	ΔI_1 (%)	ΔI_2 (%)	LT_1 (ps)	LT_2 (ps)	ΔLT_1 (ps)	ΔLT_2 (ps)	MLT	FIT
EUROFER 97-475C-100h	42.1	49.3	2.3	2.4	100	181	0	12	163.411	1.0934
MA956-475C-100h	43.1	54.7	1.2	1.2	83.5	233.9	2.5	6.1	180.8206	1.0458
PM2000-475C-100h	56	42.5	2	2.1	99.7	227.7	3.7	7.1	164.9707	1.0523

Table 7 PALS results of 475 °C aged samples for 500 h, MLT is the positron mean lifetime given as weighted average of three lifetime values. Annihilation in the air is not present in this table.

Sample	I_1 (%)	I_2 (%)	ΔI_1 (%)	ΔI_2 (%)	LT_1 (ps)	LT_2 (ps)	ΔLT_1 (ps)	ΔLT_2 (ps)	MLT	FIT
MA956-475C-500h	53.66	44.64	0.34	0.33	100	249.6	0	3.2	189.90144	1.0208
PM2000-475C-500h	57.43	41.546	0.089	0.086	100	239.31	0	0.9	172.19873	1.0053

Table 8 PALS results of 650 °C aged samples for 500 hours, MLT is the positron mean lifetime given as weighted average of three lifetime values. Annihilation in the air is not present in this table

Sample	I_1 (%)	I_2 (%)	ΔI_1 (%)	ΔI_2 (%)	LT_1 (ps)	LT_2 (ps)	ΔLT_1 (ps)	ΔLT_2 (ps)	MLT	FIT
MA956-650C-500h	49.77	48.74	0.52	0.52	100	243.3	0	3	192.90642	1.0663
PM2000-650C-500h	28.6	68.2	0.93	0.93	67	219.7	3.6	3.3	198.6934	1.085

Acknowledgement

This work was financially supported by Scientific Grant Agency of the Ministry of Education of Slovak Republic and the Slovak Academy of Sciences VEGANo. 1/0477/16 and VEGA No. 1/0204/13.