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Study of residual stress and vacancy defects in oxide dispersion strengthened steels

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Abstract

This study was focused on commercial oxide-dispersion strengthened (ODS) steels - MA 956 (20%Cr), ODM 751 (16%Cr) and ODS Eurofer (9%Cr), developed for fuel cladding of GEN IV reactors. The ODS steels are described in order to comparison their microstructure features. Vacancy defects were observed by Doppler Broadening Spectroscopy (DBS) and Positron Annihilation Lifetime Spectroscopy (PALS). Residual stress proportional to all kinds of defects was investigated by Magnetic Barkhausen Noise (MBN) measurement.

The positron techniques demonstrated the highest defect concentration for ODS Eurofer followed by MA 956. The lowest defect density belongs to ODM 751; although these defects are the largest (three or four vacancy clusters). MA 956 and ODS Eurofer have di-vacancies in predominance. MBN results are in a good accordance with positron techniques. The highest residual stress is for ODS Eurofer, followed by MA 956. Finally, the lowest residual stress proportional to hardness is found for ODM 751.

Keywords: ODS steels, Positron Annihilation Lifetime Spectroscopy, Doppler Broadening Spectroscopy, Magnetic Barkhausen Noise

1. Introduction

The design and introduction of future nuclear reactor systems is strongly dependent on choice of structural materials. These materials must withstand more stringent operating conditions, typical for reactors within the international program Generation IV (Gen IV). In compare to current commercial reactors, all construction materials (particular the internal components) of the GEN IV reactors will be operated at elevated temperatures (up to 900 -

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950^{o}C in case of High Temperature Reactors). They will be also loaded by higher radiation damage (up to tens of dpa for internal structure) due to high fast neutron flux [1].

The knowledge about ageing-induced changes in term of microstructure of construction materials [2, 3] is not sufficient for Gen IV reactors. Therefore, structural materials (new materials of Gen IV included) have to be tested and the results of these tests need to provide microstructural interpretation.

The presented paper is focused on a group of Oxide-Dispersion Strengthened (ODS) materials, which have been considered as candidate materials in most Gen IV concepts. Although the presented ODS steels were primarily designed for application in space and for heat exchangers in thermal power plants, their derivates have been developed for nuclear applications. ODS materials are appropriate for applications requiring a higher temperature limit than classical ferritic steels. ODS steels are able to resist high thermal strain up to a temperature ~ 900^{o}C [4, 5].

Our experiments provide the microstructural characterization of chosen ODS steels using non-destructive technique - Positron Doppler Broadening Spectroscopy (DBS), Positron Annihilation Lifetime Spectroscopy (PALS) and Magnetic Barkhausen Noise (MBN) techniques.

2. Experiment

Three following commercial materials - candidates for Gen IV fuel cladding were measured:

i) MA 956 (20% Cr, product of Incoloy),
ii) ODM 751 (16% Cr, product of Plansee),
iii) ODS Eurofer (9% Cr, product of Plansee).

The chemical composition of investigated steels is listed in Table 1.

Table 1 Chemical composition of steels (in % wt.).

	C	Mn	Ni	Cr	Mo	Ti	Al	Si	W	Y_2O_3
MA 956	0.07	0.12	0.07	19.94	0.10	0.30	3.40	0.04	-	0.50
ODM 751	0.07	0.07	0.02	16.20	1.74	0.70	3.80	0.06	-	0.50
ODS Eurofer	0.10	0.44	-	8.73	0.01	-	-	0.01	1.10	0.30

The investigated ODS alloys were produced by mechanical alloying, i.e. matrix materials were milled and mixed together with yttrium particles to form solid solutions with a uniform dispersion of oxide nano-particles. Then the mixture were consolidated using Hot Extrusion (ODS Eurofer) or Hot Isostatic Pressing (MA 956 and ODM 751). The MA 956, ODM 751 and Eurofer ODS alloys were supplied as rods in recrystallized condition for improvement of the creep strength. The recrystallization heat treatment resulted in a coarse columnar grain structure (the grain size ~ mm).

Samples of investigated steels were prepared from as-received material by cutting the steel sheets into suitable pieces. After cutting, the sample surfaces were polished in order to remove surface impurities (up to ~ 0.3μm).

The applied technique – Doppler Broadening Spectroscopy (DBS) [6] utilizes the principles of momentum conservation during positron annihilation [7]. The momentum of annihilating positron-electron pairs is detected as broadening of the 511 keV annihilation peak [8], which is recorded by two HPGe detectors in a single counting mode. The DBS technique expresses results in S and W parameters. The W parameter (the area of the tails of the spectra) describes annihilation with high momentum core electron. The S parameter from the peak area is typical for positron annihilating with low momentum electrons (mostly valence ones). The energy windows for our DBS measurements were 3keV<|E#-511 keV|<7.6 kev for S parameter and |E#-511 keV|<0.83 keV for W parameter.

The S and W parameters are sensitive to annihilation surroundings. The S parameter increases for the lattice with an increase of defect concentration. The W parameter decreases at the same time but with smaller gradient [6, 8]. If sample contains only bulk and one type of defects, the S (W) parameters should lie on a line between these two states [8].

The DBS was measured with a slow positron beam [9 - 11] accelerating positrons up to 36 keV. The defect depth profile can be observed in surface and subsurface layers of the samples with applying of this equipment. The maximal investigated depth slightly exceeds 1.6 μm according to calculation published in [12].

The Positron Annihilation Lifetime Spectrum (PALS) technique [13, 14] can determine concentration and size of vacancy-type defects in sample with very low concentration (from 0.1 to 500 ppm) [14]; therefore it can describe the area where transmission electron microscopy (TEM) is not so sensitive. Positron lifetime is proportional to defect size. The percentage of positrons trapped and annihilated in the defects (positron intensity), can give information about defect concentration.

As positron source, $+ radioactive ^{22}Na was used in sandwich arrangement together with 2 samples. The measuring equipment used in this work consists of two BaF_2 scintillation detectors and two discriminators [15]. The measured spectra were evaluated by program LifeTime9 [16] according to the Standard trapping model [17]. The value of FWHM parameter, which describes the resolution of the measuring equipment, was close to 240 ps. Fit variance (reduction of chi-square) achieved value in range (1; 1.1), which means that the deviation of the fit was sufficient and the deviation of the fit was below 0.1%.

Magnetic Barkhausen Noise (MBN) technique is useful for determination of residual stress in material, which relates to the presence of defects such as precipitations, grain boundaries and lattice defects. MBN is based on a magnetic field going through ferromagnetic material. During the measurement, the structure composed of domains starts to reorganize according to the external field. This phenomenon requires movement of the domains which is made by discontinuous jumps recorded as MBN signal [18]. Defects located in the samples act as pinning sites for magnetic domains and hinder the movement of magnetic domains. This tends to reduce the noise signal; therefore specific types of defects or residual stress can be observed with applying of this technique.

Results acquired by this experimental technique can be given as follows [19]:

i) Barkhausen Noise Amplitude (BNA) which characterizes a maximum value of the voltage in the MBN signal.
ii) Corresponding magnetic field (H_{peak}) describing the position of BNA in applied field.
iii) Signal envelope. The envelope is plotted as a function of the applied magnetic field (current).

BNA and the trend of the envelope are decreasing with growth of defect concentration, residual stress or hardness [18]. The position of BNA peak - H_{peak} can establish grain size as was published in [20].

The MBN measurement was performed with using of commercial system Stresstech AST %Scan 500 [21] at magnetizing frequency up to 50 Hz and a magnetizing voltage 3 Vpp (Volts peak-to-peak). The sampling frequency was set up to 1 MHz. The signal of the pick-up coil was filtered from 5 to 500 kHz and amplified with a gain of 10.

3. Results & Discussion

DBS results are acquired from annihilation spectra via S and W parameters. Figure 1a shows results in S-W diagram measured for positron implantation energy up to 36 keV. The behavior of S-W parameters differs significantly for individual samples. The lowest gradient is for ODS Eurofer, followed by MA 956. The highest gradient was found for ODM 751. This sample should have values the closest to bulk values (defect-free structure), although its values are placed only in region of defects.

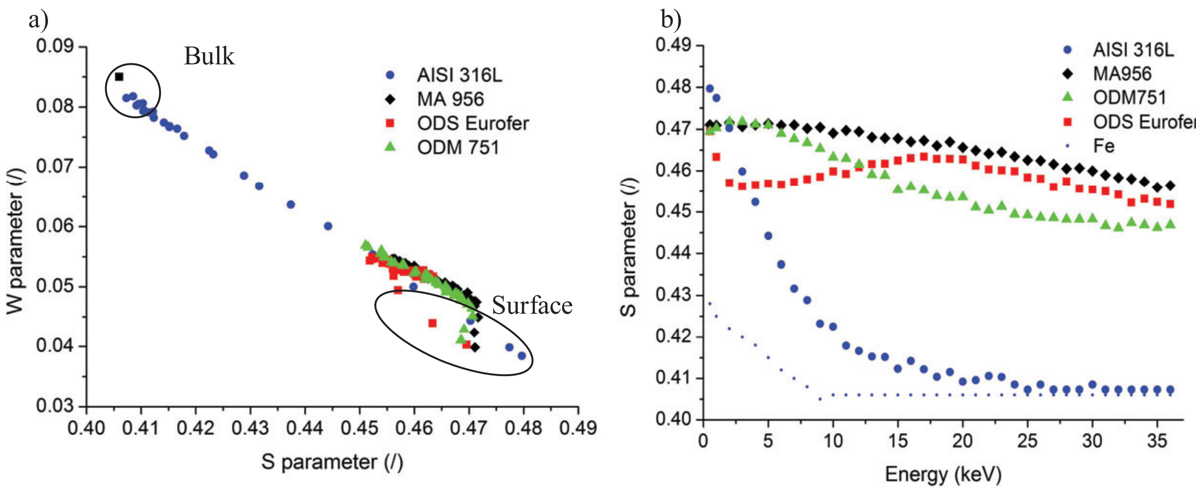

Figure 1 Behavior of W - S parameters (a); S - Energy diagram (b), relative deviation of the S and the W parameter did not exceed 0.5%.

Figure 1b represents diagram of the S parameter versus positron implantation energy up to 36 keV. The effect of positron back-diffusion in ODS steels is not visible, it means that surface and first subsurface layers (up to 5 - 10 keV) are considerably affected by polishing. In compare to ODS steels, reference sample – AISI 316L, which was polished by the same way, was probably more resistant to that. DBS results indicate that ODM 751 is described by the lowest defect concentration.

PALS measurement was applied for three different pieces of all samples. Each spectrum was treated into three lifetimes (LT). The average values are further presented.

The shortest lifetime achieved values – up to 100 ps, which describes positron annihilation in defect-free structure reduced due to defect presence according to the Standard trapping model [21]. The second positron lifetime (LT2) within the range 200 and 250 ps characterizes vacancy type defects and is proportional to defect size. The last lifetime (>500 ps) is not further mentioned, because it only corresponds to in-flight or surface annihilation as well as annihilation in source, which was not fully removed during process of source calibration.

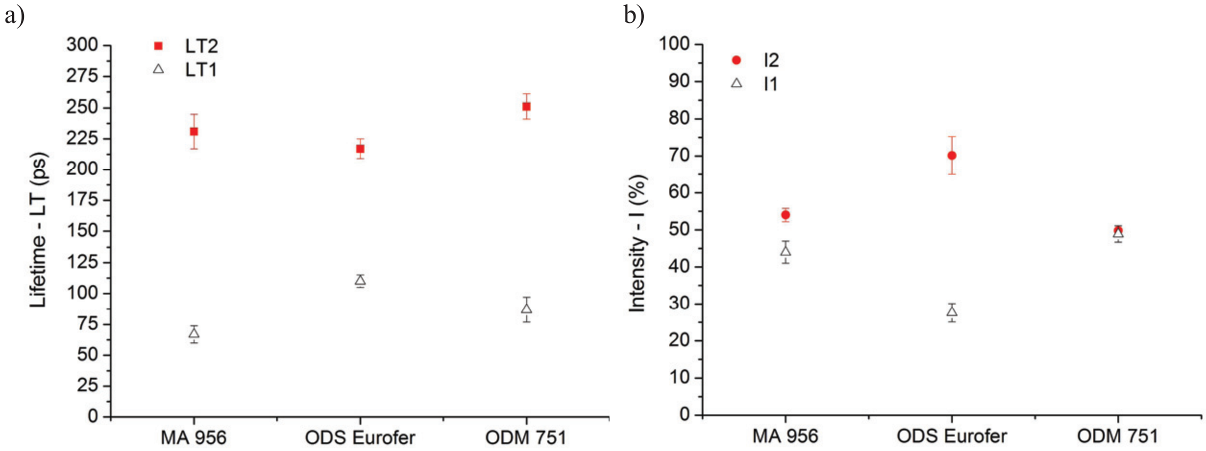

Figure 2 Results of Positron Annihilation Lifetime Spectroscopy: Lifetimes (a); Intensities (b).

The LT2 for steel MA 956 and ODS Eurofer are almost the same. MA 956 achieved 210 ps and ODS Eurofer – 218 ps (see Figure 2). ODM 751 had visible higher values – 250 ps. After comparison of measured lifetimes to theoretical values of defects [23], this signifies that MA 956 and ODS Eurofer contain defects probably with the size of di-vacancies in predominance (theoretical value ~ 200 ps), but they probably have also some three-vacancies. ODM 751 has combination of three-vacancies (theoretical value ~ 232 ps) and four-vacancy clusters (theoretical value ~ 262 ps).

The intensities (percentages) of positron annihilation in the defects (I2) differ significantly for investigated steels, i.e. for MA 956 ~ 60%, ODM 751 - 51% and ODS Eurofer – over 70%. Observed defects are categorical and they are formed during manufacture of the materials.

Mean lifetime (MLT) calculated as average value from all lifetimes referring to appertaining intensities was the highest for ODS Eurofer (205 ps), followed by ODM 751 (180 ps). Steel MA 956 contains the smallest defects and its MLT is also the smallest – 173 ps.

The MBN data are presented by BNA values, according to which the presence of defects, residual stress or hardness for each material, respectively, can be compared. The MBN results are graphically illustrated by the signal envelope. Moreover, shift of H_{peak} for individual samples is shown for purpose of comparison of grain size.

The BNA parameters are listed in Table 2. The highest signal amplitude - BNA was observed for ODM751, which demonstrates the lowest concentration of all structural defects (vacancies, precipitations, grain boundaries) than in ODS Eurofer and MA 957. It can also denote lower hardness or lower level of residual stress in ODM 751. The highest residual stress belongs to ODS Eurofer (Table 2). The smallest grains relative to smallest H_{peak} were found in MA 956. The highest H_{peak} as well as the coarsest grains were detected for ODM 751.

Figure 3 also demonstrates visible higher envelope of ODM 751, followed by for MA 956 and the smallest envelope is for ODS Eurofer.

Table 2 Results of Magnetic Barkhausen Noise measurements - Barkhausen Noise Amplitude (BNA) and BNA position (H_{peak}).

Sample	BNA (V)	H_{peak} (%)
MA 956	5.9	-25
ODM 751	14.8	16
ODS Eurofer	3.1	-3

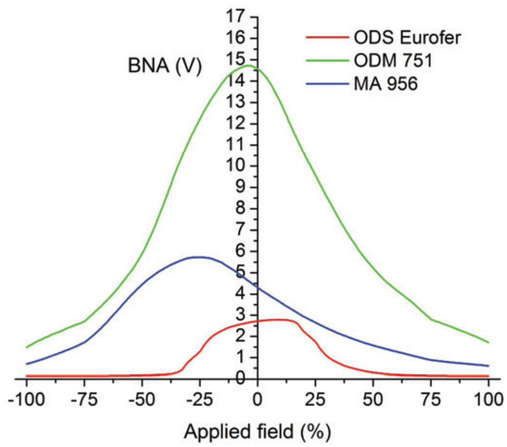

Figure 3 Signal envelope of Magnetic Barkhausen noise for frequency up to 50 Hz (depth ~ 1 mm).

Although presented experimental techniques do not observe the same defects in samples, they all can give similar information about defect concentration. DBS technique detects vacancy defects up to depth of 1.6 μm, while second positron technique – PALS is not so sensitive in this small depth. PALS described defects in the layers up to 120 μm. MBN technique can detect not only vacancy defects but also structural defects such as precipitates, grain boundaries and impurities. Thus, this study can give more complex view of defect presence in investigated materials.

The results from all applied techniques manifested that the smallest defect concentration and residual stress is for material with the largest defect size – ODM 751 (16% Cr), which was treated by Hot Isostatic Pressing. The Eurofer ODS with 9% Cr treated by Hot Extrusion is probably the hardest material which can be due to presence of precipitation as well as high concentration of vacancy defects (di-vacancies in predominance).

The differences in microstructure of the samples were caused due to difference of chemical composition. The chromium content is variable in samples, but it is not the only effect on microstructure. The observed behavior for both positron techniques and the magnetic results did not show a dependency on chromium content.

There are other influences, i.e. different process of mechanical alloying – ODS Eurofer (Hot Extrusion) versus Hot Isostatic Pressing of MA 956 and ODM 751. Also the thermal treatment and post-treatment annealing can affect microstructure.

4. Conclusion

Positron annihilation lifetime spectroscopy, Doppler broadening spectroscopy and Magnetic Barkhausen Noise measurements were applied in the microstructural study of the commercial Oxide Dispersion Strengthened steels – MA 956, ODM 751 and ODS Eurofer. Differences in microstructure of the studied materials were found, which resulted from small variance of chemical composition, manufacturing and probably post-manufacturing thermal treatment. Our assumption, that chromium content can play important role in defect presence as precipitations and even in open volume defects, was not confirmed. The ODS Eurofer with the smallest chromium content contains the highest defect concentration, which can be also due to differences in mechanical alloying during manufacture in compare to other investigated ODS steels.

In further work, the comparison of the microstructural features to mechanical properties will be studied.

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References

[1] S. J. Zinkle, Fusion Materials Science: Overview of Challenges and Recent Progress, APS Division of Plasma Physics 46 Annual Meeting, Savannah, GA, USA (2004).
[2] C. W. Hunter, G. D. Johnson, Mechanical Properties of Fast Reactor Fuel Cladding for Transient Analysis, ASTM STP 611 (1976) 101. ISSN 0066-0558.
[3] R. L.Klueh et al., J. Nuc. Mat. 341 (2005) 103.
[4] B. A. Pint. et al. J. Nuc. Mat. 307-311 (2002) 763.
[5] R.Lindau, A. Möslang, M. Rieth et al., ODS-Eurofer – A reduced activation feritic martenistic ODS-steels for application in advanced blanket concepts, Proc. of EUROMAT congress, Prague (2005). Retrieved from http://www.extremat.org/ib/site/publication/downloads/Paper%20Lindau1.pdf
[6] S. Eichler, R. Krause-Rehberg, Appl. Surf. Sci. 149 (1999) 227.
[7] R. Krause-Rehberg et. al., Appl. Phys. A 66 (1998) 599.
[8] J. Slotte et. al., Phys. Rev. B 78 (2008) 085202.
[9] K. Saarinen et. al., Semiconduct. Semimet. A 51 (1998) 210.
[10] J. Oila et. al., Appl. Surf. Sci. 194 (2002) 38.
[11] J. Lahtinen et. al., Nucl. Instr. Meth. Phys. Res. B 17 (1986) 73.
[12] M. J. Puska, P. Sanku, R. M. Nieminen, J. Phys. Condens. Matter. 1 (1989) 608.
[13] V. Sluge , V. Kršjak, M. Petriska, A. Zeman, M. Mikloš, Physica Status Solidi C 4 (2007) 3481.
[14] V. Sluge , What kind of Information we can Obtain from Positron Annihilation Spectroscopy, Report, DG JRC, Institute for Energy, European Commission, Netherlands, 2006. EUR 22468.
[15] M. Petriska, A. Zeman, V. Sluge , V. Kršjak, S. Sojak, Phys. Stat.Solidi. C 60 (2009) 2465.
[16] J. Kansy, Nucl. Instr. Meth. Phys. Res. A 374 (1996) 235.
[17] P. Hautojärvi, C. Corbel, Positron spectroscopy of solids, Amsterdam, IOS Press, 491, 1995. ISBN 90 5199 203 3.
[18] X. Kleber, A. Hug-Amalric, J. Merlin, Metalurg. & Mater. Trans. A 39 (2008) 1308.
[19] M. Willcox, T. Mysak, An introduction to Barkhausen Noise and its Applications. [online] Insight NDT. Available from: www.insight-ndt.com/papers/technical/t013.pdf [cited 2010 22 Sept.
[20] R. Baldev, T. Jayakumar, V. Moorthy, S. Vaidyanathan, Russian Journal of nondestructive Testing 37 (2001) 789.
[21] Informational website of StressTech Group, [online] Stresstech group. Available from: http://www.stresstech.fi [cited 2010 5 Sept].
[22] Y. C. Wu, Y. Q. Chen, B. Wang, S. J. Wang, Y. C. Jean, R. Suzuki, T. Okdaira, Appl. Surf. Sci. 252 (2006) 3274.
[23] P. Hautojärvi, Positrons ed. Solids, Springer, Berlin, 1979, p.89-144.