THE EFFECTS OF HIGH TEMPERATURE HELIUM GAS ON THE FRACTURE BEHAVIOUR OF ODS_MA957

ROZUMOVÁ Lucia[1], HOJNÁ Anna[1], KALIVODOVÁ Jana[1], BUBLÍKOVÁ Petra[1], HADRABA Hynek[2], DI GABRIELE Fosca[1]

[1]Research Centre Rez, Hlavní 130, Husinec-Řež, 250 68, Czech Republic

[2]Institute of Physics of Materials of the Academy of Sciences of the Czech Republic, v.v.i., Žižkova 22, Brno, 616 62, Czech Republic

Abstract

Oxide-dispersion-strengthened steel (ODS) MA957 has been studied as a candidate material for Tritium Breeding Blanket for fusion reactors with helium coolant, where temperatures 250–650 °C are expected. Helium is one of type as primary coolant in High Temperature Reactor and Gas Cooled Fast Reactor with working temperature 500-1000 °C.

This work presents results of the study of behaviour of ODS (MA957) under influence of high temperature helium environment. Microstructure of the material and the interaction with He was investigated. The average exposure temperature was held at 720 °C. Subsequently, the surface changes were determined. Impact testing and material surfaces were studied in detail by means of SEM and TEM microscopy. It is essential to understand the He effect on microstructure and mechanical properties of structural materials since the He embrittlement in TBB may be significant.

Keywords: ODS, MA957, Helium

1. INTRODUCTION

With the development of GEN IV reactors, also materials have to be developed. The foremost consideration in the successful development and deployment of GEN IV reactor systems is the performance and reliability issues involving structural materials. The structural materials need to endure much higher temperatures, extremely corrosive environment and neutron doses. Materials for use in different reactor components include various FM steels, austenitic stainless steels, nickel-base super alloys, ceramics, composites, ODS steel, etc. FM, ODS and austenitic stainless steels are considered as structural materials in almost all GEN IV systems [1]. However, it is also important to remember that some desirable characteristics for the Gen-IV structural materials are noted below:

- Excellent dimensional stability against thermal and irradiation creep, void swelling,
- Favorable mechanical properties such as strength, ductility, creep rupture, fatigue, creep–fatigue interactions,
- Acceptable resistance to radiation damage (irradiation hardening and embrittlement) under high neutron doses (10–150 dpa or displacements per atom), helium embrittlement,
- High degree of chemical compatibility between the structural materials and the coolant as well as with the fuel [2].

Oxide dispersion strengthening (ODS) is excellent material candidates for use in fusion reactor plants. Oxide dispersion-strengthened (ODS) steels are being developed and investigated for nuclear fission and nuclear fusion applications in Japan, Europe, and the United States.

ODS steels have excellent creep strength, corrosion and radiation resistance. Application of ODS steels in these advanced nuclear systems with huge and complex structures as well as suitable bonding and welding techniques need further development. These techniques must provide such a process that the microstructures with very fine grains and homogeneous distribution of nano-scaled oxide particles are not remarkably changed by the joining processing [1].

The MA957 steel contains small amount (about 0.25%) nano-sized yttrium-rich particles. Yttrium-rich increases creep strength of the steel and effectively suppress softening annealing by blocking dislocations motion at elevated temperatures and subsequent recrystallization of ferritic matrix of the steel. Two main problems are connected to the toughness of these type steels: transitional behavior of ferritic matrix of the steel and strengthening of the steel by oxide dispersion. Increase in the yttria content led to use at the higher temperatures. Long-term exposure at elevated temperatures of the MA957 steel led to by migration of dislocations and subsequent recrystallization of elongated ferrite grains to the equi-axed ones. The process of recrystallization led to the outstanding loss of strength and toughness of the steel. [6, 7, 8].

The higher coolant temperature in combination with other system parameters (pressure, flow, neutron flux in the reactor core) results in higher demands on resistance of structural materials. Therefore the high temperature, corrosion resistant materials need to be developed and validated.

The aim of the work was to investigate the impact of temperature in helium on specimens of ODS steel MA957 and describe differences in microstructure in order to assess the changes of material.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

The MA957 steel of nominal composition (wt.%) Fe–14Cr–0.9Ti–0.3Mo–0.25Y2O3 was produced by mechanical alloying process [3]. This steel contains small amount (about 0.25%) homogeneously dispersed nano-sized yttrium-rich particles for increasing creep strength of the steel [4]. The chemical composition of both alloys is presented in Table 1. The MA957 was manufactured by IPM ASCR, v. v. i. from the commercially available atomized iron powder, ferrotitanium powder, ferrochrome powder, yttria powder and molybdenum powder. Those powders were mixed in exact proportions and processed in high energy ball mills for 24 hours in the air atmosphere. Afterwards the degassed powder was cold pressed to compact pellet and rod of diameter 30 mm was hot-extruded at 1150 °C from the pellet [5].

Table 1 Chemical composition of MA957 [wt. %]

Elements	**Cr**	**Ti**	**Mo**	**Y_2O_3**	**Fe**	**Al**	**C**
MA957	13.6	0.85	0.29	0.25	Bal.	0.06	0.007

2.2. Experimental - Exposure in Helium

The high temperature corrosion tests were carried out under atmospheric pressure using the gas mixtures described in Table 2. Specimens of MA957 were exposed to the flowing gas (0.25l/min) in a quartz tube reactor inserted in a high temperature furnace. Specimens were heated up at a rate of 4 C/min then maintained at 720°C. Duration of the exposure test was 500 hours. In order to avoid any presence of oxygen in the furnace, a container with Ti foam was placed in the quartz tube and used as an oxygen getter. After exposure the weight changes were determined.

Table 2 Chemical composition of the gaseous mixture

Impurity	CO_2	O_2	CH_4	CO	H_2	He
Concentration [ppm]	1	2	35	250	400	Bal.

The chemical composition of both specimens, before and after exposure was analyzed by SEM/EDX scanning electron microscopy SEM (LYRA3. VEGA3, Tescan) and also TEM (JSM 6460, Jeol). Chemical composition of the steel was measured by means of emission spectral analysis (Spectrumat GDS 750, Leco).

Mini-Charpy KLST specimens of 3 × 4 mm cross section and length of 27 mm were machined according to the DIN 50115 standard oriented in uniaxial direction (L-T orientation). An instrumented impact testing of specimens prepared was conducted in the temperature region between -180°C to +24°C according to the standards EN 10045-1, ISO 148 and EN ISO 14556. The microstructure of the steel was observed by means of optical microscopy, scanning electron microscopy (JSM 6460, Jeol) and transmission electron microscopy (CM12, Philips).

3. RESULTS AND DISCUSSIONS

The influence of the high temperature helium environment resulted in oxidation of the surface of MA957 specimens. The average weight gain after 500h exposure was. 0.6 mg/cm^2. An accurate description of the oxide layer as well base material was obtained by combining SEM and TEM analytical techniques.

The microstructure of MA957 steel contained extremely fine and rounded grains, (Fig. 1). The diameter of the grains of MA957 was about 500 nm.

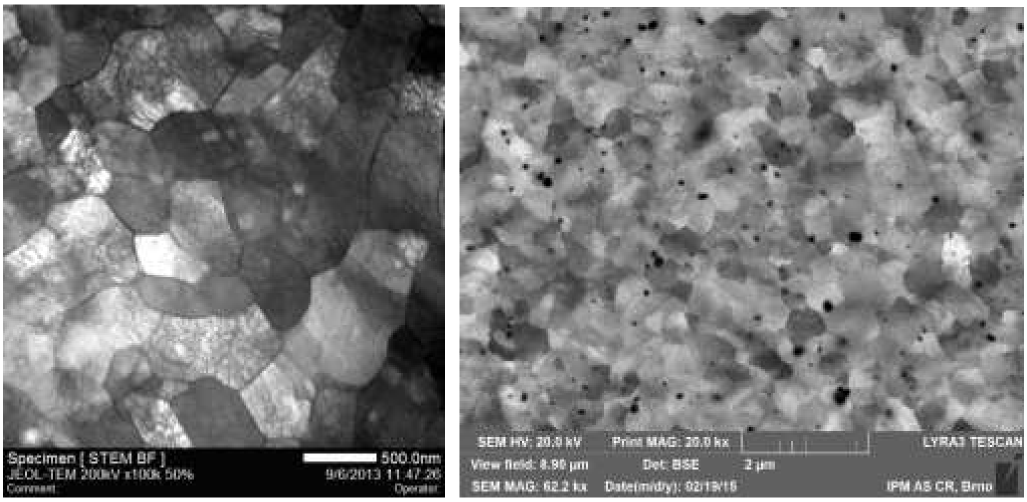

Fig. 1 Micrograph of the oxide dispersion in MA957 before (left) and after (right) the exposure in helium.

Nanosized dispersed particles was proven by means of SEM/EDS analysis and majority of longer particles were of yttrium–titanium–oxide type.

The temperature dependence of impact energy of MA957 steel measured in the temperature region from −180 °C to + 24.5 °C is given in the Fig. 2. The lower shelf energy (LSE) region of the impact energy was about 0.5 J and upper shelf energy (USE) region was about 10.5 J.

Fracture surface are complex and affected by texture of the material. Brittle fracture of samples fractured at LSE was formed by cleavage micro mechanism.

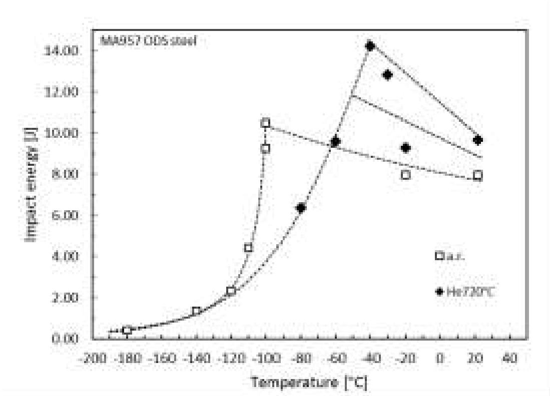

Fig. 2 Temperature dependence of impact energy of MA957 steel in as-received state and after the exposure helium environment

Ductile fracture of samples broken at USE was formed by dimple (micro-void coalescence) micro mechanism. The fracture surfaces contained also high amount of cracks oriented perpendicularly to the fracture surface [5].

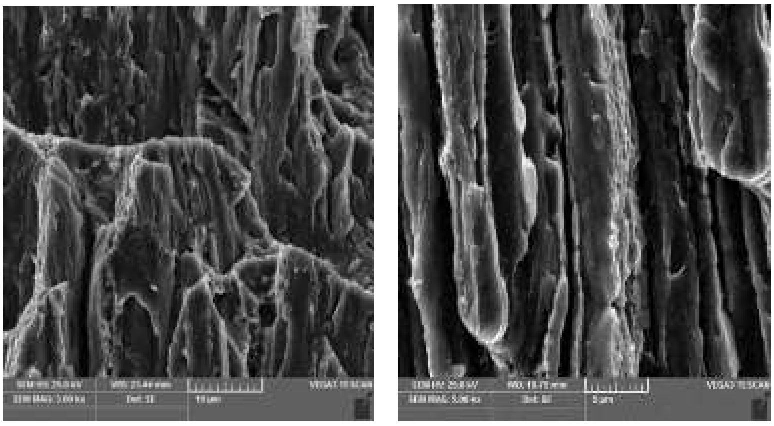

Fig. 3 Cleavage fracture micro mechanism of MA957 ODS exposed to He 720˚C and impact tested at -80˚C (left) and -30˚C (right)

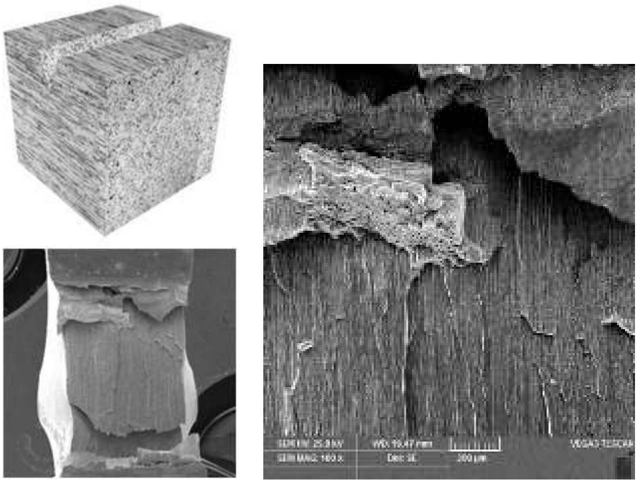

Fig. 4 The 3D reconstruction of texture MA957 and fracture surface of mini Charpy specimen exposed to He 720˚C and impact tested at -80˚C

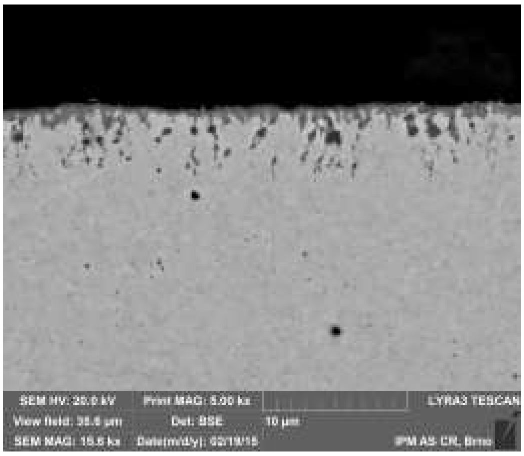

Fig. 5 Surface of MA957 ODS after exposure in He at 720 °C for 500h

Fig. 5 shows the cross-sectional morphology of MA957 specimen after exposure test in the high temperature helium. The internal oxidation of the specimen surface can be observed. The thickness of oxidation layer was about 2μm. The connection along the oxidation layer/alloy substrate interface was not compact. Cracks and voids in the contact areas can be observed defect-rich or even porous oxide scale will be a less efficient diffusion barrier compared to a lattice with low defect concentrations. EDS analysis indicated that the oxide layer was mainly Ti-enriched oxide layer. Rather not satisfactory oxidation resistance of MA957 steel was attributed to the high temperature, but also to the steel manufacture. The migration of elements towards surface of the material will have further negative effect also on mechanical properties of the material. Apart from that, several voids in the base material can be seen in Fig 6. Therefore bigger attention shall be paid to manufacturing process development of the ODS steel.

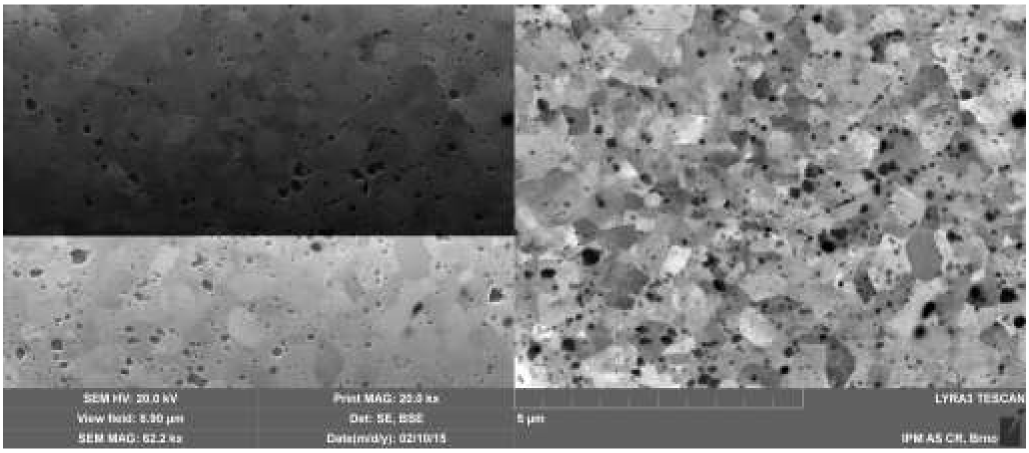

Fig. 6 MA957 after exposure in He: showing grains in transversal direction and stable pores

CONCLUSION

The MA957 steel prepared by mechanical alloying process and subsequent one step thermo-mechanical treatment – hot extrusion at 1150 °C was studied. Specimens have been exposed to high temperature helium environment. MA957steel does not possess an excellent oxidation resistance at the high temperature. After exposure in helium at 720℃ for 500 h, the thickness of oxidation layer was about 2 μm and the oxidation layer does not possess protective characteristics. The material embrittlement owing to the He exposure resulted in transition temperature shift about 40˚C.

ACKNOWLEDGEMENTS

This work has been supported by the SUSEN Project CZ.1.05/2.1.00/03.0108 realized in the framework of the European Regional Development Fund (ERDF) and by the GACR project No. 14-25246S.

REFERENCES

[1] BARTOSOVA I., VETERNIKOVA J., SLUGEN V. Study of candidate materials for new reactor systems using positron annihilation spectroscopy and Barkhausen noise. Nuclear Engineering and Design, Vol. 273, 2014, pp. 376-380.

[2] MURTY K.L., CHARIT I. Structural materials for Gen-IV nuclear reactors: Challenges and opportunities. Journal of Nuclear Materials, Vol. 383, 2008, pp. 189-195.

[3] FISCHER J.J. US Patent 4075010, No. 21, February 1978.

[4] ALAMO A., LAMBARD V., AVERTY X., MATHON M.H. Assessment of ODS-14%Cr ferritic alloy for high temperature applications. Journal of Nuclear Materials, Vol. 329–333, 2004, pp. 333–337.

[5] HADRABA H., KAZIMIERZAK B., STRATIL L., DLOUHY I. Microstructure and impact properties of ferritic ODS ODM401 (14%Cr-ODS of MA957 type). Journal of Nuclear Materials, Vol. 417, 2011, pp. 241-244.

[6] ALINGER M.J., ODETTE G.R., LUCAS G.E. Tensile and fracture toughness properties of MA957: implications to the development of nanocomposited ferritic alloys. Journal of Nuclear Materials, Vol. 307–311, 2002, pp. 484-489.

[7] YANG W.J., ODETTE G.R., YAMAMOTO T., MIAO P., ALINGER M.J:, HRIBERNIK M., LEE J.H. A critical stress–critical area statistical model of the KJc(T) curve for MA957 in the cleavage transition. Journal of Nuclear Materials, Vol. 367-370, 2007, pp. 616-620.

[8] PREININGER D. Modelling of the effect of precipitates on work-hardening, ductility and impact behaviour of ferritic–martensitic Cr steels. Journal of Nuclear Materials, Vol. 307–311, 2002, pp. 514–520.