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Conference Paper

Structure and Crystallographic Texture Changes of Ferritic Martensitic Steel Resulting from Thermal Creep and Ageing Tests

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Abstract

Thermal ageing (650 and 700°C during 1000, 7000 or 13300 h) and creep (700°C, 50 MPa) tests of tubes made from ferritic-martensitic steels EK181 and ChS139 were carried out. With the aid of X-ray techniques the investigation of crystallographic texture and structure condition after tests was conducted. Thermal ageing provides substructure enhancement. With the increase of ageing time one can note the decrease of microhardness and X-ray peaks broadening, which indicates inner elastic microstress relaxation. It was revealed that changes of crystallographic texture in the rupture area of steel ChS139 tube after creep test is similar to those after uniaxial tensile test at room temeprature. This indicates the similarity of the mechanisms of grain reorientation for creep and tension. Recrystallization occurs in steel EK181 during creep test at temperature 700°C leading to formation of recrystallization texture. This results in faster failure of steel EK181 (2486 h before rupture) in comparison with steel ChS139 (3426 h).

1. INTRODUCTION

Ferritic-martensitic steels are perspective materials for the cladding tubes in future fast breeder reactors [1]. Among of the most significant characteristics of cladding tube material are the ability to conserve their properties at high temperature during the long time and at applied load. In this work a study of structure, texture and mechanical properties of tubes made from ferritic-martensitic steels EK181 and ChS139 were carried out after their ageing and creep tests.

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2. MATERIALS AND METHODS

Cladding tubes of ferritic-martensitic steels (composition is in table 1) have a diameter of 6.9 mm and the wall thickness of 0.4 mm. Initial state of these tubes was as follows: heat treatment at 1190°C for 25s and then at 720°C for 2h [1].

Table 1: Chemical composition of steel EK181 and ChS139.

Steel	C	Cr	Mn	Mo	Nb	V	W	Ni	N	Si	P	Ti	B	Zr
EK181	0.15	11.17	0.74	0.01	0.01	0.25	1.13	0.03	0.04	0.33	0.010	–	0.006	0.05
ChS139	0.21	11.85	0.57	0.51	0.30	0.31	1.26	0.73	0.085	0.29	0.007	0.01	0.006	0.01

Thermal ageing tests were carried out at temperatures 650 and 700 °C during 1000, 7900 and 13300 hours. Specimen preparation for the analysis was cutting of tube segments of size 3x1 along the tube axis *L* (fig. 1, a), their grinding, polishing and electrolytic etching in a mixture of phosphoric acid $H_3PO_4$and chromic anhydride CrO_3 at voltage 24V and current value 2.4 A. Prepared segments were attached together on a substrate forming a compound square sample of size 3x3 as in fig. 1, b.

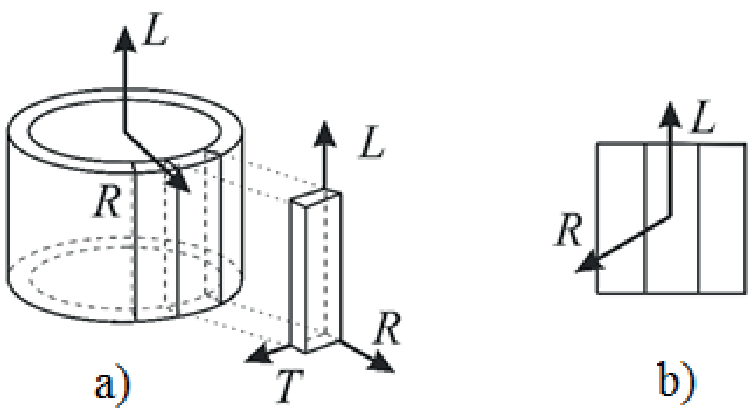

Figure 1: Sample preparation scheme for analysis of aged tubes.

Uniaxial creep tests were carried out at temperature 700 °C and load 50 MPa without special atmosphere (on air) until rupture. Steel EK181 fractured after 2486 hours and steel ChS139 after 3426 hours. On Figure 2 relative diameter change of tubes along their length after creep tests is shown (length count starts at rupture place). After the distance of 48 mm the zone of thermal influence origin from a welding attachment to the holders starts, which is of no interest for this work.

For X-ray texture analysis a small rings (of 3 mm height) were cut out of the tubes. Place and number of zone, out of which the cutting was performed are pointed out on fig. 2 by red dotted lines. Further preparation of samples consisted of electrolytic

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etching of surface in a mixture of phosphoric acid $H_3PO_4$and chromic anhydride CrO_3 at voltage 24V and current value 2.4 A.

For texture investigation a direct pole figure measurement technique (DPF) was used [3]. X-ray texture measurements were conducted on diffractometer DRON-3 using chromium radiation while X-ray spectra were obtained on diffractometer Bruker D8 Discover using copper radiation. Mechanical properties were estimated by microhardness measurement, obtained using the analysis of indentation curves [4].

3. EXPERIMENTAL RESULTS

3.1. Ageing tests

On Figure 3 are shown the graphs of full width at half maximum (FWHM) of X-ray lines (222) and (200) versus ageing time for ageing tests of steels EK181 and ChS139 at 650 and 700°C.

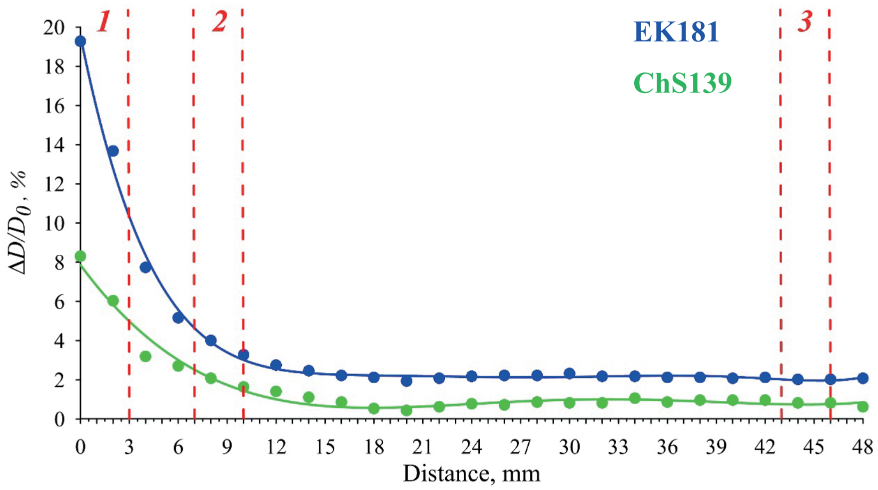

Figure 2: Relative diameter change of tubes made of EK181 (blue line) and ChS139 (green line) steels along the distance from the rupture place after their creep test.

On Figure 4 are shown changes of microhardness versus ageing time for tests of steels EK181 and ChS139 at 650 and 700°C. Reference points indicating the microhardness in the initial state of tubes were added on the graphs at the ageing time of 0 h.

On Figure 5 DPFs {100} are shown for steels EK181 (a, b) and ChS139 (c, d) in initial state (a, c) and after ageing at 700°C for 13300 h.

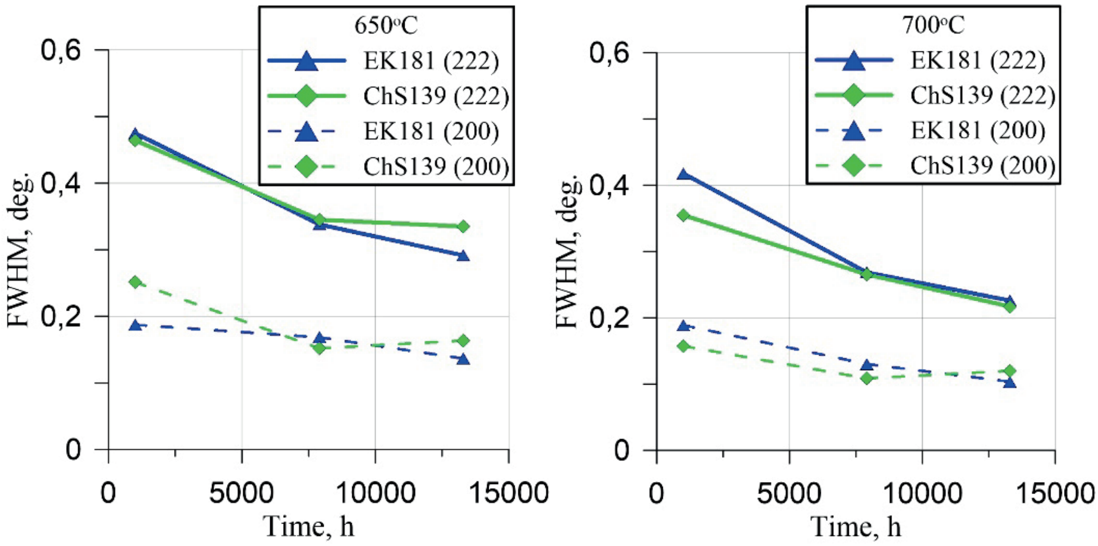

Figure 3: FWHM of lines (222) (solid line) and (200) (dotted line) for steels EK181 (blue) and ChS139 (green) after ageing tests at 650 (a) and 700°C (b) versus ageing time.

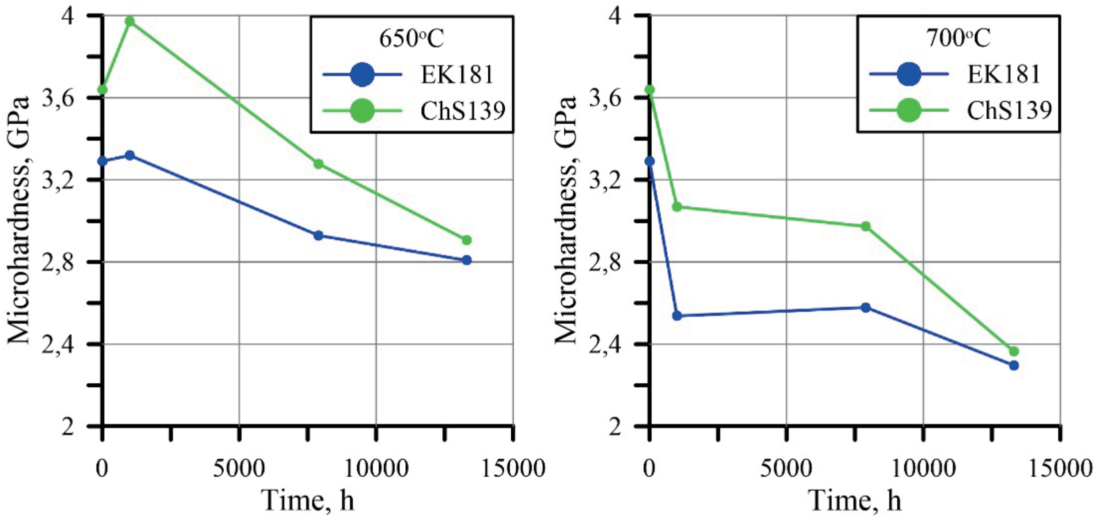

Figure 4: Microhardness changes versus ageing time of steel EK181 (blue) and ChS139 (green) at 650 (a) and 700°C (b).

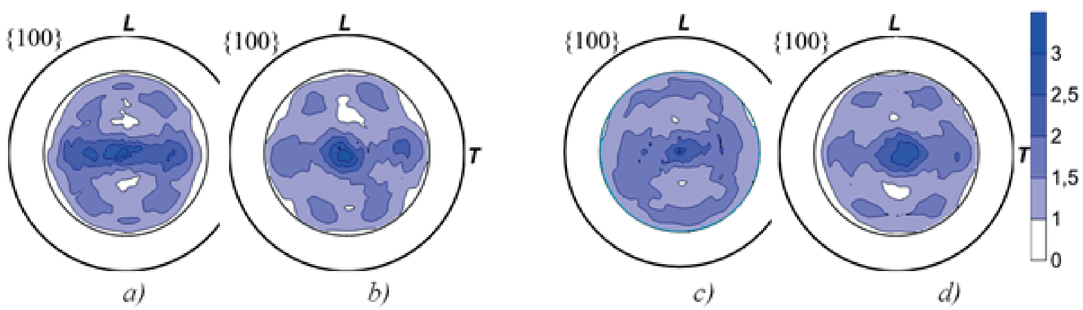

Figure 5: DPF {100} for steels EK181 (a,b) and ChS139 (c,d) in initial state (a,c) and after ageing at 700°C for 13300 h (b,d).

3.2. Creep tests

On Figure 6 are shown DPFs {110} and {100} for steels EK181 (a) and ChS139 (b) measured from samples in initial state (A), from fracture zone 1 (B) and from the zone 3, distant to fracture area (C). Color scale is the same, as in fig. 5.

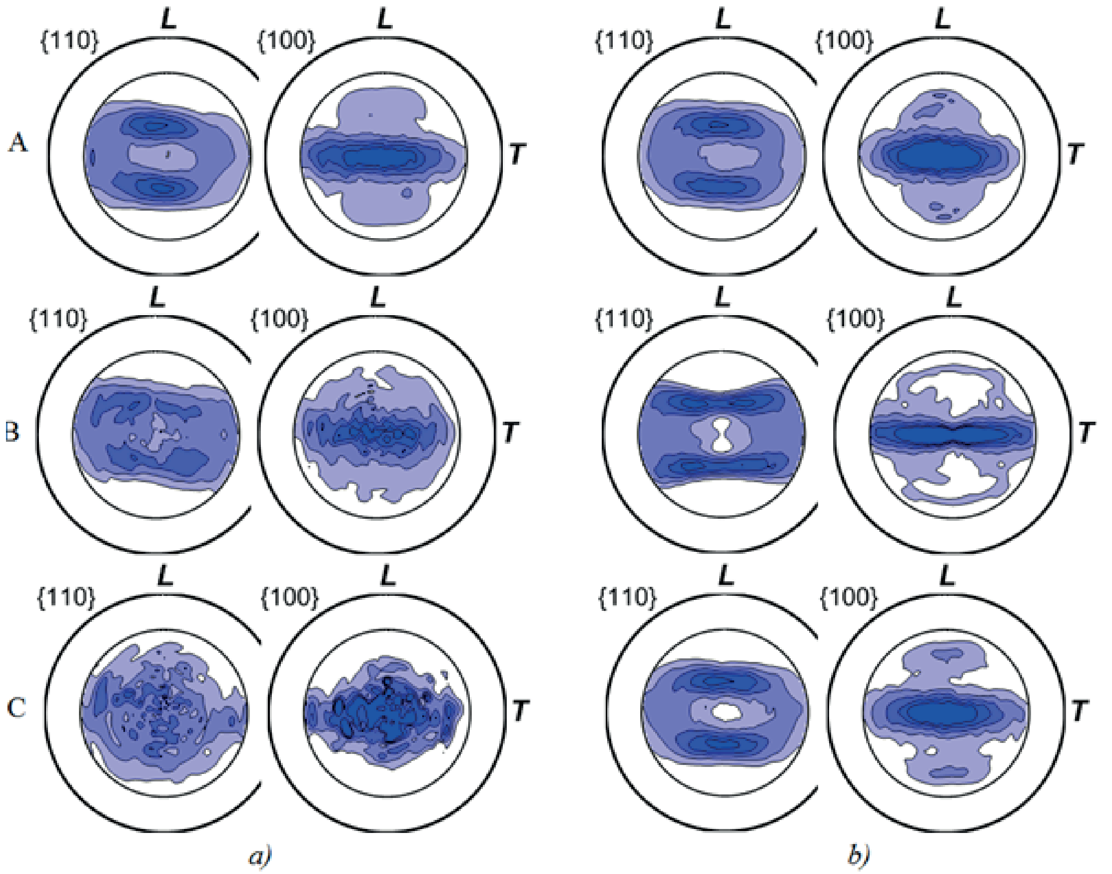

Figure 6: DPFs {110} and {100} for steels EK181 (a) and ChS139 (b) in initial state (A) and after creep test: rupture area (B) and area, distant from rupture zone (C).

4. Discussion

Ageing of steels EK181 and ChS139 leads to the structure improvement, characterized by the decrease of X-ray peaks width (fig. 3). In the works [5-7] there is a similar behavior of structure changes (decrease of dislocation density, increase of subgrains size, decrease of microdeformation of lattice) with the time increase. According to fig. 3 the decrease of broadening of lines (222) and (200) for the steels EK181 and ChS139 at different temperatures is almost similar, which indicates an identical rate of structure recovery.

Along with the structure improvement in studied steels the decrease of microhardness is observed (fig. 4). At this, steel ChS139 is characterized by higher values of microhardness, regarding the steel EK181 at different ageing times. It is worth noting that at the ageing time of 1000h and at temperature 650°C there is an increase of microhardness, while at 700°C – a decrease of microhardness. Further microhardness changes are similar for different ageing times and different temperatures.

According to [6] after ageing of steels EK181 and ChS139 a coarsening of carbides of type $Me_{23}C_6$, where Me – Cr, Fe, Mo etc., is observed. It was also stated that the size of carbides of type Me(C, N), where Me – V, Ti, Nb etc., is relatively stable at temperatures not higher than 620°C. Initially concentration of carbides mentioned above is higher in steel ChS139, which fact can explain the higher value of microhardness in initial tube ChS139 regarding steel EK181. At ageing process with similar conditions (620°C, 13000h) in steel ChS139 $Me_{23}C_6$ carbides are noticeably bigger, than in steel EK181. Although structure relaxation process is similar in both steels (fig. 3), the process of movement and rearrangement of atoms, which leads to coarsening of carbides and changing a matrix chemical composition, can exhibit different kinetics in different steels (because of different chemical composition, see table 1) and at different temperatures [5-7].

According to DPF at fig. 5 the texture of steels EK181 and ChS139 do not undergo any radical changes, i.e. structure recovery does not include reorientation processes. It indicates that in studied steels after ageing no recrystallization in the bulk of samples is present. There is a possibility of polygonization process, since the structure improvement is established (fig. 3). Worth noting, that even ageing at temperature 700°C for 13300 hours does not lead to recrystallization.

Texture changes during creep test in steel ChS139 (fig. 6, b) shows that in this steel an ordinary mechanisms of plastic deformation are induced during a creep deformation. The enhancement of texture components {111} <110> and {112} <110> observed, which is analogous to the uniaxial tensile deformation. At the distance from the rupture, texture is similar to that of initial sample, indicating the absence of deformation or recrystallization.

After creep test of steel EK181 (fig. 6, b) in the distance from the rupture (fig. 6, B) one can notice a smoothed fractional texture, indicating the recrystallization in the bulk of sample (the smoothing and fractioning is a result of disturbance in measurement, given by coarsened and reoriented recrystallized grains). In the fracture area there is a tendency to form texture components {100-111} <110>, which are characteristic for the deformation texture.

According to the ageing tests, the sample of steel EK181 aged for 13300 hours at temperature 700°C did not have a recrystallization (fig. 4), while in creep tests at temperature 700°C lasting for 2486 hours there is a significant recrystallization, manifested in texture smoothing and fractioning. It appears that additional load, applied to the tubes at creep test, shifts the temperature of recrystallization start below the 700°C. The recrystallization effect observed in tests is deteriorative because it decreases the stability of ferritic-martensitic tubes, since the tube of steel EK181 have much more rapid failure, than tube of steel ChS139 without the recrystallization.

5. CONCLUSIONS

1. Ageing of steels EK181 and ChS139 results in structure improvement, characterized by reducing of X-ray lines broadening. At that, crystallographic texture indicates absence of the recrystallization in the bulk.

2. As a result of creep test of steel ChS139 a reorientation of the grains takes place according to regularities analogous to uniaxial tensile test. In the rupture area texture changes are the most pronounced, while in the distance from rupture texture is similar to that in the initial tube.

3. In steel EK181 after creep test a recrystallization texture is observed, partly disturbed in the rupture area due to plastic deformation. Additional load appears to aid recrystallization start, since additional energy reduces temperature of recrystallization beginning.

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