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Changes of structure and crystallographic texture of cladding tubes from austenitic steel under thermal creep testing

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Abstract. The process of changes in structure and crystallographic texture of cladding tubes from austenitic steel ChS68 under thermal creep testing were studied. Testing of tubes was conducted at the temperature 700 ^{o}C in the air by their stretching in axial direction under the stress 160 MPa until rupture. By data of phase and texture analysis a number of processes, accompanying plastic deformation of tubes during thermal creep tests at elevated temperature, were identified. The main texture components of original tube, as well as texture components of different parts of the tested tube are {110}<001> and {112}<11$\bar{1}$>. In the rupture zone the component of the texture of tension with axis <111> along the tube axis becomes stronger. This effect is connected with activation of dislocation slip in the deformed area of tested tube near the new-formed neck. At the same time the character of texture changes in the zone of tube rupture indicates to development of the dynamic recrystallization, conditioned by the total influence of all factors, which control the passage in the tube of thermal creep. In addition, it was revealed the activation of martensitic transformations in the zone of maximal deformation of tube as a result of its creep tests.

1. Introduction

The service period of a fast breeding reactor fuel rods is largely determined by the creep of the cladding material, since its upper part is known to have the highest temperature. Therefore it is necessary to provide the required values of rupture strength and creep rate, which are conditioned by the stability of structure and phase composition of the material during the period of operation. To provide stability of structure it is necessary to conduct systematical study of creep mechanisms of cladding tubes from austenitic steels. Crystallographic texture is sensitive indicator of the processes activated during plastic deformation. Examination of the changes of texture and phase composition along the tested tube reveals features of the processes that are manifested in the high-temperature tests, such as phase transformations, plastic deformation and dynamic recrystallization.

In the current work the regularities of structure and texture changes of cladding tubes from austenitic steel ChS68 as a result of their creep test were studied.

2. Experimental

Cladding tubes 6.9x0.4 mm from ChS68 steel (composition is given in table 1) were examined as follows:

1. Initial state: austenization, 20% cold work (sample 1);
2. Half of a tube after rupture during uniaxial (along the axis of the tube) creep test (sample 2).

Testing proceeded in the air at a tensile stress of 160 MPa, a temperature of 700 °C; time to failure - 6028 hours. According to the measurement of the tube elongation during the test total strain amounts 1.6%. By the measurement of the tested tube diameter (figure 1), minimum residual strain amounts to 0.3%, whereas in the rupture zone strain amounts 2%.

Table 1. Chemical composition of ChS68 steel.

Composition, wt. %													
C	Si	Mn	S	P	Cr	Ni	Mo	Ti	V	B	N	Co	Al
0.05	0.5	1.7	0.006	0.005	16.4	14.8	2.0	0.26	0.1	0.003	0.005	0.01	0.02

Oxide film of the tested tube was removed by 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. The thickness of the removed layer is approximately 30-35 µm. X-ray structure analysis and phase analysis were performed for the etched surface of the tested tube. To conduct the study of the crystallographic texture for both initial and tested tube the small rings with height of 3 mm were cut out from the tubes on the electrospark discharge machine. Red lines that points out the cut and red numbers of the corresponding rings used for the texture measurement are placed on figure 1.

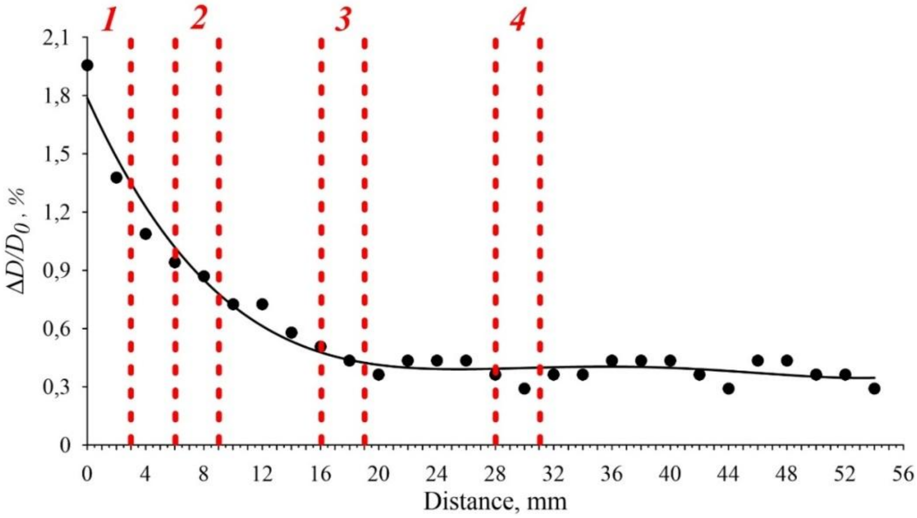

Figure 1. Relative diameter change of cylindrical sample 2 along tested tube. Count starts from the destruction place.

3. Methods

In the current work following methods of structure and texture study were used:

1) X-ray phase analysis method;
2) methods of construction of direct and inverse pole figures.

The investigation of the structure of austenitic tubes was conducted on diffractometer D8 DISCOVER (Bruker, Germany) using filtered radiation CuK_{α}. For the phase identification Bruker AXS DIFFRAC.EVA v.3.0 software and ICDD PDF-2 database were used.

Quantitative phase analysis of the samples from austenitic steel was not carried out because of the presence of crystallographic texture, which causes significant redistribution of the intensity of diffraction spectrum lines.

The study of crystallographic texture is usually conducted by X-ray measurement of direct pole figures (DPFs) {hkl}, using the tilting of sample [1]. DPF {hkl} is a distribution of a pole density of the selected type <hkl> normal in a stereographic projection of the tube sample with its characteristic external axes: a radial *R*, tangential *T* and longitudinal *L*. Pole density is directly proportional to the intensity of the X-ray beam reflected from the respective planes {hkl}. Therefore the registration of the intensity of X-ray beam diffracted from the planes {hkl} for all possible sample orientations relative to the directions of the primary and reflected beams should be conducted for the measurement of DPF. Planes of the selected {hkl} type are reflecting at different orientations of sample. Normal to this planes divides the angle between the primary and reflected beams in two. The amount of the intensity registered is proportional to the volume of grains, taking part in diffracted beam formation.

Texture measurement was conducted on DRON-3 diffractometer, equipped with automatic texture set, with the accumulation of data on the hard drive of a personal computer. Further treatment was carried out with the use of the original software. Incomplete DPF {100}, {110} and {111} were constructed, permitting to determine main texture components of austenitic phase.

Inverse pole figures (IPF) show probable distribution of external axis of interest belonging to the sample relative to the internal crystallographic axis. Plane normal is attributed to the weight proportional to the probability of matching them to the axis of the sample [1].

Direct and inverse pole figures are special cases of orientation distribution function (ODF) [2]. With the use of mathematical instruments needed it is possible to obtain IPFs from ODF. In this paper, the construction of the IPFs carried out by their calculation of ODF, restored on incomplete DPFs using LABOTEX software [3]. This prevented the preparation of texture free reference sample.

4. Results

4.1. Phase analysis results

According to [4] the thickness of the oxide layer on surface of the cladding tubes from austenitic steel, tested under similar conditions does not exceed 7-10 μm. Diffraction spectra were recorded for all fragments of the tube, indicated on figure 1 by red numerals. Recording was performed after removing oxide film from the surface of the tube by etching. Spectra were used to identify phase composition of the samples. Following phases were found:

- γ-phase of the iron, austenite;
- traces of Fe_2O_3 oxide;
- α′-martensite (base centered tetragonal structure) and ε-martensite (HCP structure).

Figure 2 shows that with increasing distance from the rupture area varies the quantitative ratio of the detected phases. The intensity of the peaks, corresponding to austenitic and martensitic phases is proportional to the composition of the respective phase. Figure 3 shows how intensity of (111) peak of austenitic phase increases (the amount of austenite increases), while intensity of (110) peak of martensitic phase decreases (the amount of martensite decreases).

Besides that on spectrum (Figure 2) one can find peaks, corresponding to oxide phase, most intense in locations of the investigated tube, where α′-martensite peaks also have the highest intensity.

Additional etching of 20-25 μm leads to the disappearance of peaks, corresponding to the martensite phase and oxides. Only austenite phase peaks remain in the spectra.

4.2. Crystallographic texture

According to the received data, texture of cold worked and annealed (tested under the temperature of 700 °C) are characterized by a set of approximately the same texture components: {110}<001> and {112}<11$\bar{1}$> (figure 4).

To observe the changes of crystallographic texture of the tested tube it is more convenient to build IPF. In this work IPF were built from DPF {100}, {110}, {111}. Figure 5 shows IPF of *R* and *L* directions of initial tube and of zones 1 and 4 of the tested tube. The appearance of new texture peaks on IPF_R of zone 1 texture near the [[122]], [[113]], [[123]] and [[013]] poles was detected.

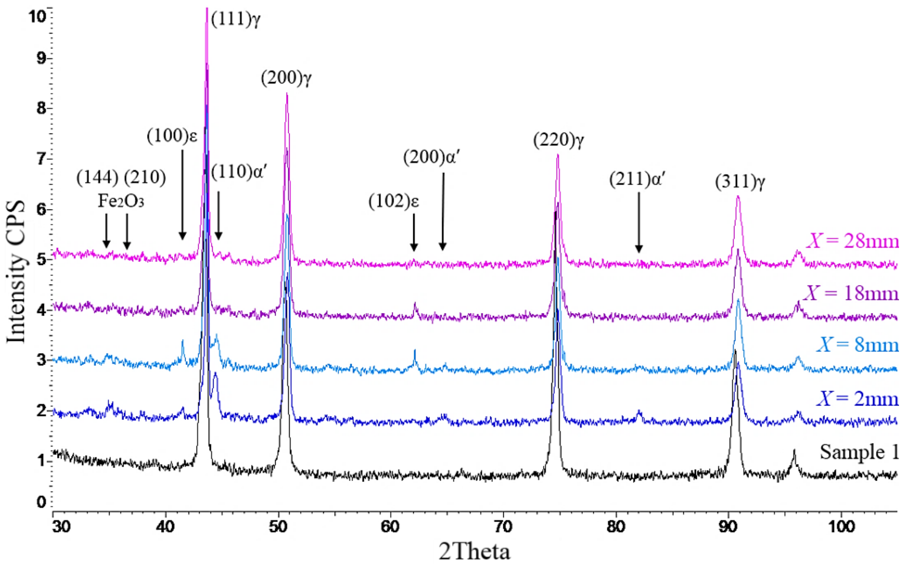

Figure 2. The spectra of different areas of the tested tube, located at various distances X from the rupture place.

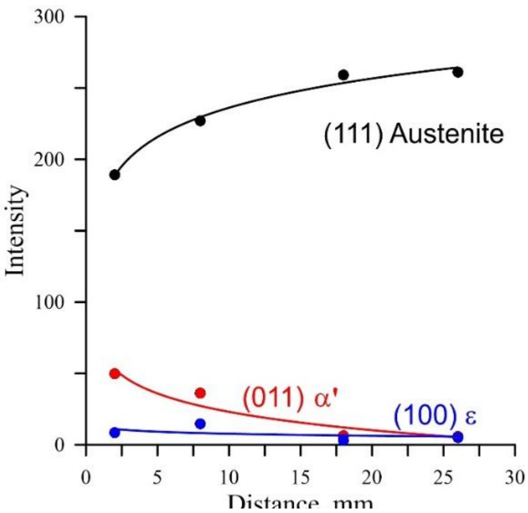

Figure 3. Intensity of the peaks (hkl), corresponding to different phases as a function of the distance from the rupture place, sample 2.

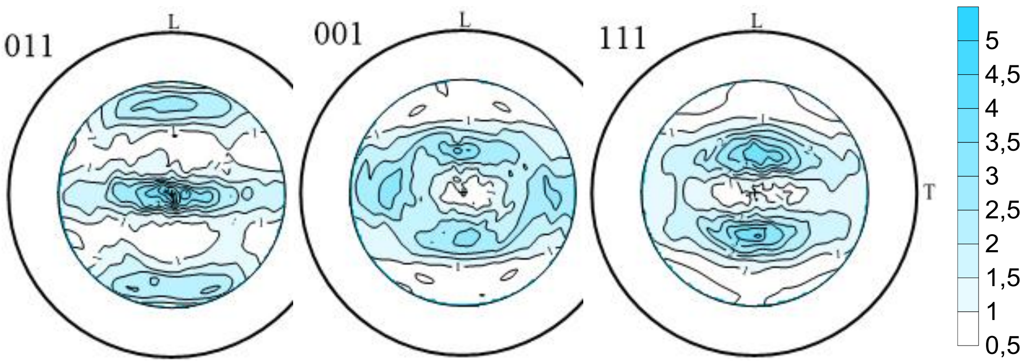

Figure 4. Incomplete direct pole figures {100}, {110}, {111} of initial tube cold worked by rolling to 20% (sample 1).

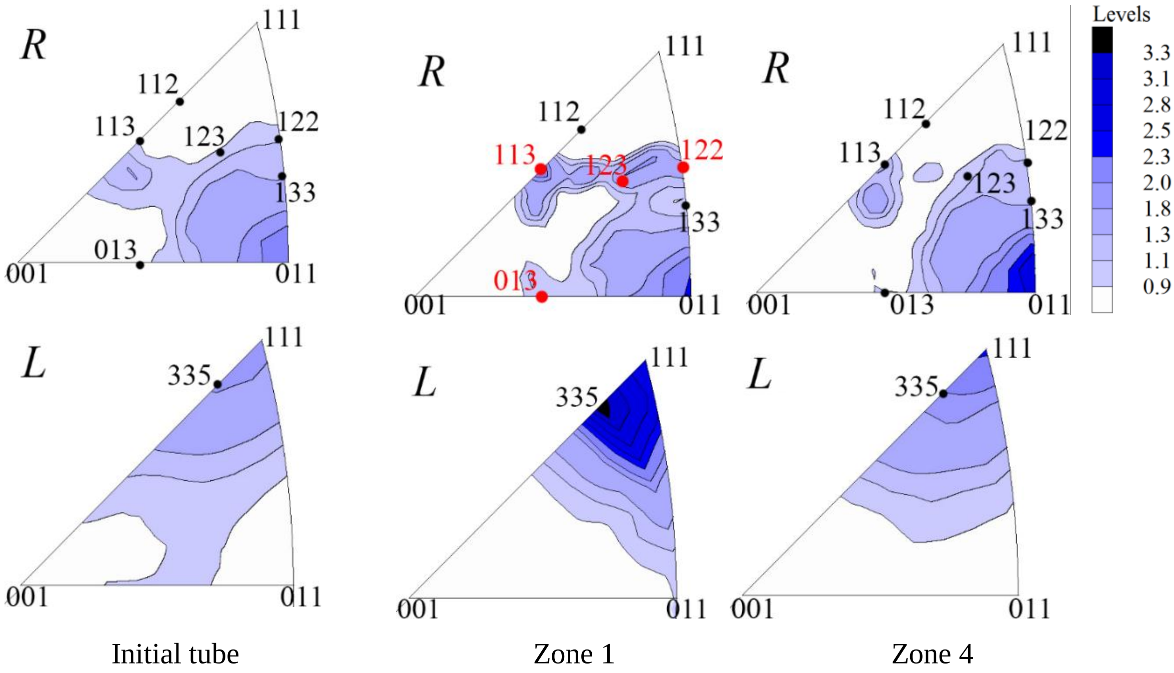

Figure 5. IPF *R* and *L* of sample 1 and of sample 2 (zones 1 and 4)

5. Discussion

5.1. Martensitic formation

There is a clear relationship between the presence of oxide film and martensite peaks on diffraction spectra of sample 2. Peaks of Fe_2O_3 oxide phase on figure 2 corresponds with presence of α'-martensite peaks.

Martensite formation in austenitic steels of different compositions was studied in works [5-7]. During the oxidation process of austenitic steels there is possible the formation of both the oxide film and the propagation of intergranular corrosion, associated with the precipitation of carbides at the grain boundaries. In both cases, the oxidation process is accompanied by a change in the chemical composition of the boundary areas (chrome depletion, oxygen saturation). This leads to the shift of martensite start temperature M_S in a higher values. As a result a martensitic formation in austenitic steels subjected to corrosion is observed.

The works devoted to martensitic transformations are pointing out that the appearance of α'-martensite is possible along with the formation of intermediate ε-martensite by the following scheme: $\gamma \rightarrow \varepsilon \rightarrow \alpha'$ [8-11]. There is a direct correlation between amounts of martensite formed and the level of deformation applied. The formation of ε-martensite is observed only in cases of stress induced martensitic transformations. The decreasing of martensite amount along the tube length starting from the rupture area, as well as the presence of traces of ε-martensite are both suggest the idea of the occurrence of stress induced martensite transformations in the studied steel. Although the absence of additional evidences puts under a question the possibility of stress induced martensitic transformations in the investigated steel.

5.2. The features of texture changes of the tested cladding tube

Initial cladding tube is characterized by FCC rolling texture {110}<001> [12]. Different parts of the tested tube was deformed up to different strain levels. For example, material of zone number 4 (red numeral on figure 1), placed far away from the rupture (28-30 mm) have suffered the lowest diametric deformation, so this zone can be used as test piece, stored at the test temperature for over than 6000 hours.

All zones of the tested tube have the same texture components {110}<001> as for the initial tube. Polygonization and aging of the tube at the test temperature provide intensification of the main rolling texture components.

The difference between the texture of the number 1 zone and the texture of other zones of the tested tube is the presence in the texture of zone 1 of additional peaks {221}, {113}, {123} and {013} (figure 5). These components are specific to the recrystallization textures of FCC materials [12]. Appearance of the mentioned components could testify about activation of the recrystallization process in the rupture area. Thus, deformation of the tube at the temperature of 700 ^{o}C promotes the formation and growth of new grains, orientation of which is corresponds to the slopes of texture maximums. This process certainly be accompanied by redistribution of alloying elements in the material of the test tubes.

6. Conclusions

Following results were obtained for the ChS68 steel creep tested at the temperature of 700 ^{o}C under the applied stress of 160 MPa:

1. Martensite formation, associated with the change in steel composition as a result of oxidation processes, was detected.
2. The change of martensite amount along the tested tube length indicates a greater thickness of the layer saturated oxygen in the rupture zone.
3. The main texture components of the initial tube and of the different zones of the tested tube are {110}<001>. In the rupture zone the component of the texture of tension with axis <111> along the tube axis becomes stronger. This effect is connected with activation of dislocation slip in the deformed area of tested tube near the new-formed neck.
4. The character of texture changes in the zone of tube rupture indicates to development of the dynamic recrystallization, conditioned by the total influence of all factors, which control the passage in the tube of thermal creep.

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