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TEXTURE AND MICROSTRUCTURE OF ANNEALED AISI302 STEELS WIRES

TEKSTURA I MIKROSTRUKTURA WYŻARZANYCH DRUTÓW ZE STALI 302

The texture and microstructure of cold-drawn and annealed wires made from AISI 302 austenitic steel were the object of the investigations. The wires were deformed up to 70.6% and 91.6% of reduction and annealed for 1 hour in the temperature range 550 – 850°C. Significant amount of martensite, formed due to the strain induced ($\gamma \rightarrow \alpha'$) transformation, were detected within the deformed structure by applying magnetic and X-ray diffraction methods. During annealing of deformed wires the reverse ($\alpha' \rightarrow \gamma$) transformation of martensite into austenite occurred.

The texture analysis of the examined wires was conducted on the basis of experimental pole figures as well as calculated inverse pole figures and orientation distribution functions. The austenite texture in cold-drawn wires was described as a strong axial texture with two <111> and <100> components. The martensite texture contained two components, the major was the <210> and the weaker one the <110> orientation. The microstructure of the deformed wire exhibited a fibre character. The structure of martensite was fine and dispersed appearing mainly within the areas of the austenite matrix displaying strain localization. In the austenite texture after annealing the same two <111> and <100> orientations remained dominant. The martensite texture weakened with increasing annealing temperature, due to the reverse transformation ($\alpha' \rightarrow \gamma$). Effects of deformation were still observed in the case of the wires annealed at lower temperatures (below 650°C). TEM investigations confirmed the presence of ultra fine martensite in the microstructure. When annealed at higher temperatures (above 650°C), the fibre microstructure was retained, but with clearly visible effects of the recovery and recrystallisation processes and the martensite was not observed any more.

Keywords: austenitic steel, wire, texture, microstructure, deformation, annealing, reverse transformation

Przeprowadzono badania ciągnionych na zimno a następnie wyżarzonych drutów ze stali austenitycznej AISI 302. Druty po odkształceniu 70.6 i 91.6% wyżarzano w zakresie temperatur 550-850°C przez 1 godzinę. Badania magnetyczne i rentgenowskie wykazały, że po odkształceniu w strukturze stali pojawia się martenzyt w efekcie przemiany indukowanej odkształceniem ($\gamma \rightarrow \alpha'$). Podczas wyżarzania zachodzi przemiana odwrotna martenzytu w austenit ($\alpha' \rightarrow \gamma$).

Analizę tekstur przeprowadzono w oparciu o figury biegunowe, trójwymiarową funkcję rozkładu orientacji FRO i odwrotne figury biegunowe. Austenit po odkształceniu posiada stosunkowo silną teksturę osiową <111> i <100>. Teksturę martenzytu odkształconego opisują dwie składowe silniejsza <210> i słabsza <110>. Mikrostruktura materiału odkształconego posiada pasmowy charakter. Martenzyt jest rozproszony, tworzy się między innymi na przecięciach pasm ścinania

Po wyżarzaniu w teksturze austenitu nadal dominują orientacje <111> i <100>. Wraz ze wzrostem temperatury wyżarzania tekstura martenzytu ulega osłabieniu co związane jest z zachodzeniem przemiany odwrotnej $\alpha' \rightarrow \gamma$. Po wyżarzaniu drutu w niższych temperaturach (poniżej 650°C) w mikrostrukturze widoczne są efekty odkształcenia. Obserwacje za pomocą transmisyjnego mikroskopu elektronowego potwierdzają obecność martenzytu. Mikrostruktura jest "super" drobnoziarnista. Powyżej temperatury 650°C martenzyt prawie całkowicie zanika. Wyżarzanie przy wyższych temperaturach (powyżej 650°C) zachowuje nadal pasmową mikrostrukturę w której widać efekty stopniowo zachodzącego zdrowienia i rekrystalizacji.

1. Introduction

Usually the major purpose of cold-working is to obtain final products with desired shape. Cold-working lead to the specific for a given material deformation structure and texture. However the ultimate mechanical properties depend on the microstructure and texture developed in the course of final annealing texture.

Rods and wires during drawing are simultaneously stretched parallel to their axes and radially compressed. Double fibrous texture <111>+<100> usually develops in the wires with FCC structure. Volume fractions

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of these two texture components differ for particular metals and alloys and depend on the SFE of materials, since the operating deformation mechanisms affect the type of the developed texture [1].

Frequently upon cold-working of stainless steel a metastable austenite transforms into martensite due to the strain induced phase transformation ($\gamma \rightarrow \alpha'$) [2, 3÷6]. In cold drawn wires produced from metastable austenitic steels, the <111> texture dominates in the γ phase. The martensite texture depends on the mechanism of phase transformation [7, 8]. In a number of works it was (ex. [2, 7]) found that in the austenitic steel of 18-8 type the major components in the γ phase were <111> while the <100> orientations were weaker. It was reported that the martensite texture was either a transition one between <320> and <210> components or included orientations of type <211>. The martensite transformation proceeded according to the Kurdjumow-Sachs (K-S) and Nishyama-Wasserman (N-W) crystallographic relation between γ and α phases.

Twinned areas and structure effects resulting from strain localization were observed in the structure of the steel. Martensite was usually formed within the structure of deformed austenite matrix for example twined areas, shear bands, etc. During annealing a specific microstructure and texture changes occur in metals and alloys. These changes are the result of several processes i.e. recovery, recrystallization, precipitation as well as the reverse phase transformation. In the metastable austenitic steels for which a strain martensite is formed upon deformation the reverse $\alpha' \rightarrow \gamma$ transformation occures during annealing [9÷16].

The final annealing texture after heat treatment results from the relative contribution of this process, affecting the final properties of the material. That is why the main purpose of the present examination was to determine the texture and microstructure after annealing at different temperatures of cold-drawn wires of metastable austenitic steel AISI 302.

2. Material and experimental procedure

The cold-drawn and subsequently annealed wires of the austenitic AISI302 steel of chemical composition given in Table 1 were the objects of the present examination.

TABLE 1

The chemical composition of the AISI 302 steel [wt.%]

C	Cr	Ni	Mn	Mo	Si	P	S	N	Fe
0.094	17.62	7.75	0.89	0.42	0.7	0.026	0.003	0.03	balance

The particulars concerning deformation schedule of the wires are given by the present authors elsewhere [17]. The AISI 302 wires deformed up to 70.6% and 91.6% of reduction were annealed at 1 hour at four different temperatures from the range 550°C–850°C.

The specimens after deformation and the subsequent annealing were investigated by means of X-ray diffraction methods. The measurements were conducted on the longitudinal sections of the wires. Qualitative and quantitative phase analyses were carried out on HZG4 diffractometer using Co Kα radiation of $\lambda_{K\alpha}$ = 0.17902 nm. Texture measurements were carried out on Bruker D8 Advance diffractometer using Co Kα radiation of $\lambda_{K\alpha}$ = 0.17902 nm.

Three incomplete pole figures – for each phase were recorded; {111}γ, {200}γ, {220}γ planes for austenite and the {110}α, {200}α and {211}α for martensite and the orientation distribution functions (ODFs) were calculated for both phases.

The metallographic optical microscope Leica 3000 N and transmission electron microscope JEM 200 MX were used for microstructure observation.

3. Experimental results and discussion

The results of the X-ray phase analysis for the initial state after deformation and subsequent annealing are presented in figure 1A and 1B. In the as-received state the 111γ, 200γ 220γ and 311γ diffraction lines were only visible on the X-ray diagrams, what indicated entirely austenitic structure of the material. After deformation the additional 110α, 220α and 211α diffraction lines from martensite appeared (Fig. 1). After 70.6% and 91.6% of cold-drawing the amounts of deformation induced martensite detected in structure were equal to 21% and 45% respectively. The results of phase analysis, texture measurement and microstructure observation of material in the as-received state and after successive deformation stages are given and discussed by the present authors elsewhere [17]. In the present examination the starting material was that after 70.6% and 91.6% of reduction.

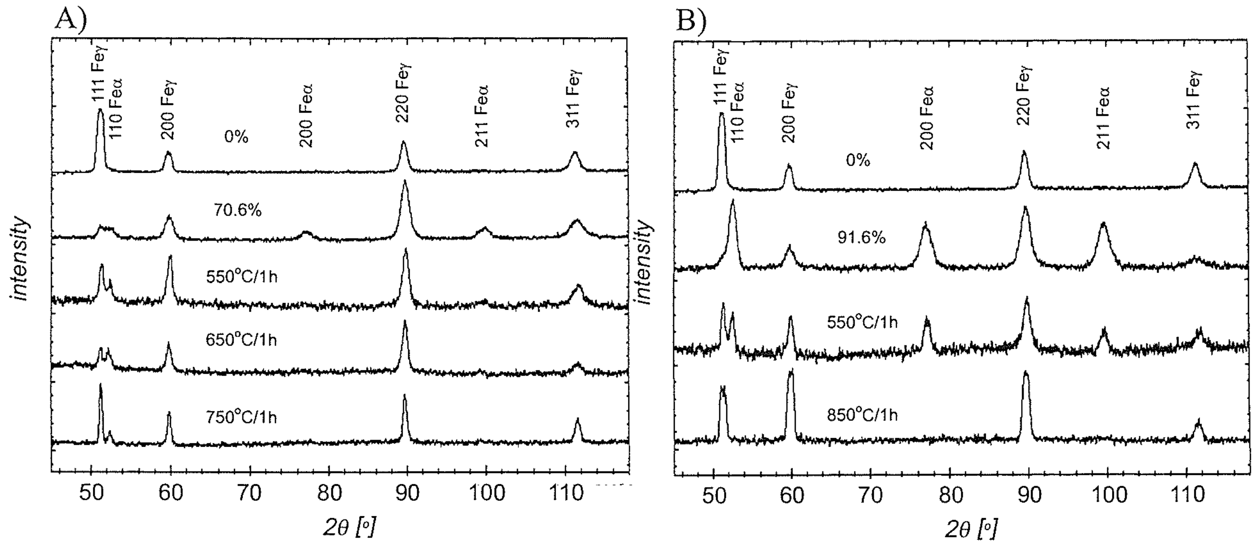

Fig. 1. X-ray diffraction patterns of the AISI 302 steel in the initial state, after deformation and after annealing at different temperature for samples deformed to 70.6% (A) and 91.6% (B)

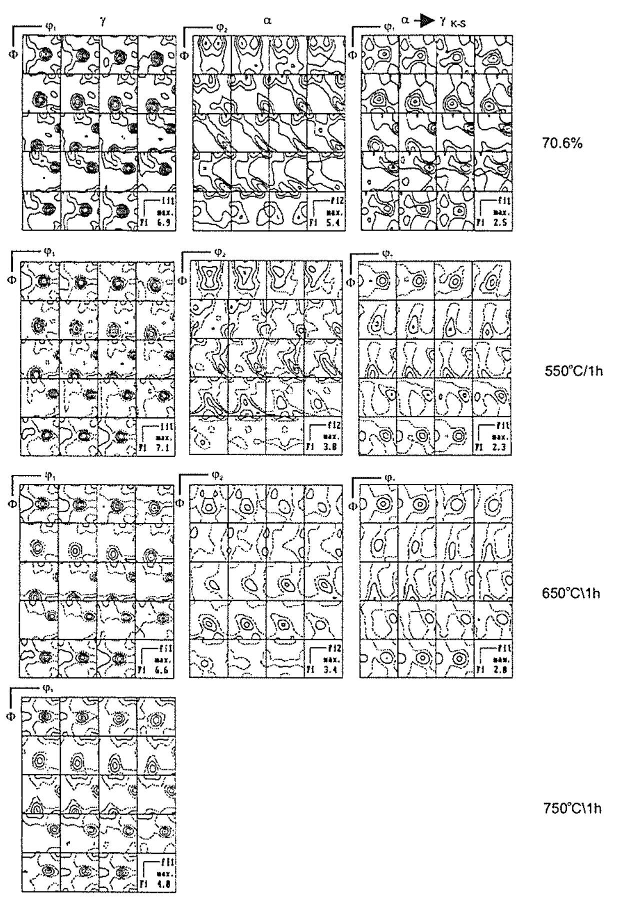

Fig. 2. Experimental austenite and martensite textures after 70.6% of deformation and annealing presented by ODF and ODF calculated for austenite (γ) from experimental α-phase on the base of assumption that K-S relation operate at inverse $\alpha' \rightarrow \gamma$ transformation

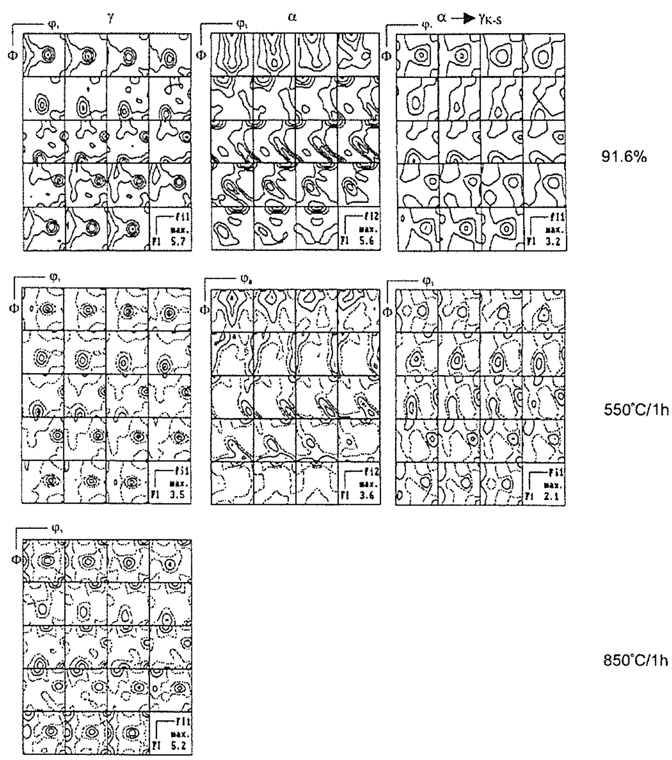

Fig. 3. Experimental austenite and martensite textures after 91.6.6% of deformation and annealing presented by ODF and ODF calculated for austenite (γ) from experimental α-phase on the base of assumption that K-S relation operate at inverse $\alpha' \rightarrow \gamma$ transformation

The results of texture measurements in the from ODF's for these two deformation stage are presented in figures 2 and 3. The resultant deformation texture comprises the texture components from austenite and strain induced martensite. Two major components the; {hkl}<111> and {0kl}<001>, in which the <111> and

<001> directions were parallel to the wire axes describe the texture of deformed austenite. The strongest components were the {011}<111> and {112}<111> orientations. The martensite texture was described by two components: the strong one <210> and the weaker one <110>. These two components of the martensite texture were transformed from those austenite texture components which belonged to the fibre α = <110>|| ND and its spread. The texture of martensite which was formed due to strain induced deformation was weaker in comparison to the texture intensity of the austenite matrix (Fig. 2). Essentially the only difference between the deformation textures of both phases after 70.6% and 91.6% of reduction was the texture intensity, that is smaller intensity of austenite texture and slightly stronger texture for martensite at higher deformation degree.

Within the microstructure in the as received state the equiaxial austenite grain with twins were visible [17]. After deformation above 70% of reduction all austenite grains were elongated parallel to the drawing direction. Depending on the used etching either grain boundaries or deformation bands were revealed (Fig 4a and 4b). Electron micrographs for the steel deformed up to 91.6% of reduction (Fig. 5) reveal strong heterogeneity of the structure in the form of shear bands. The areas with high density of thin parallel deformation microtwins were observed in transmission electron microscope foils. (Fig. 5). TEM diffractions taken from areas with different dislocation density confirmed the K-S relation between adjacent areas of the austenite and martensite. (Fig. 5).

The deformation effects were still observed in the microstructure of samples annealed at lower temperatures (550–650°C) in the form of deformed bands and the fibrous morphology paraller to the axis direction (Fig. 4c). After annealing at higher temperature (850°C), the austenite grains of different size were observed indicating the development of the recrystallisation (Fig. 4d). The electron micrograph taken after 91.6% deformation and annealing at 550°C reveal ultrafine microstructure consisting of two component phases, that is austenite and martensite (Fig. 6).

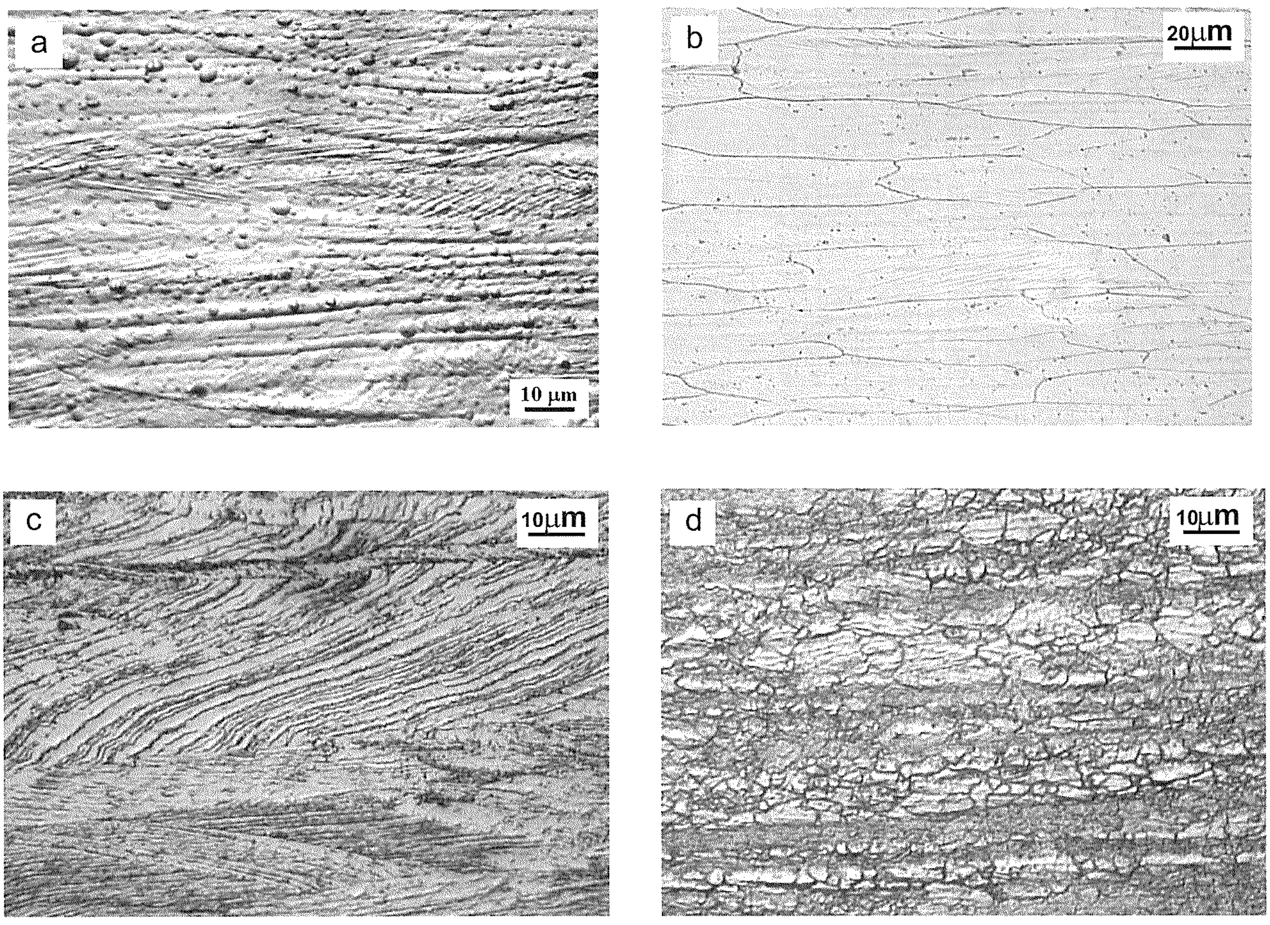

Fig. 4. Optical microstructure of the AISI 302 steel after 70.6% of deformation (a and b) and after annealing at 650°C and 850°C for 1 hour (c and d – respectively)

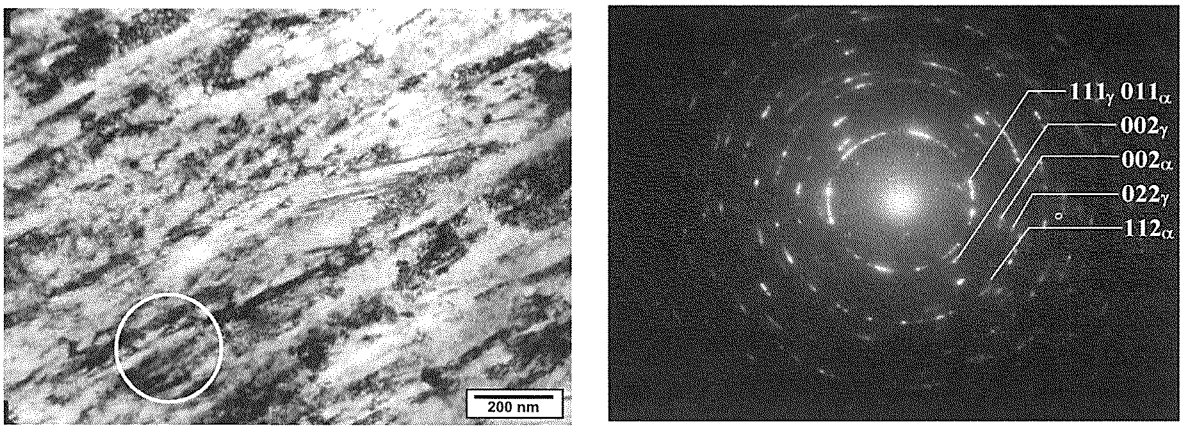

Fig. 5. TEM microstructure of the AISI 302 steel after 91.6% of deformation and SAED taken from the marked area

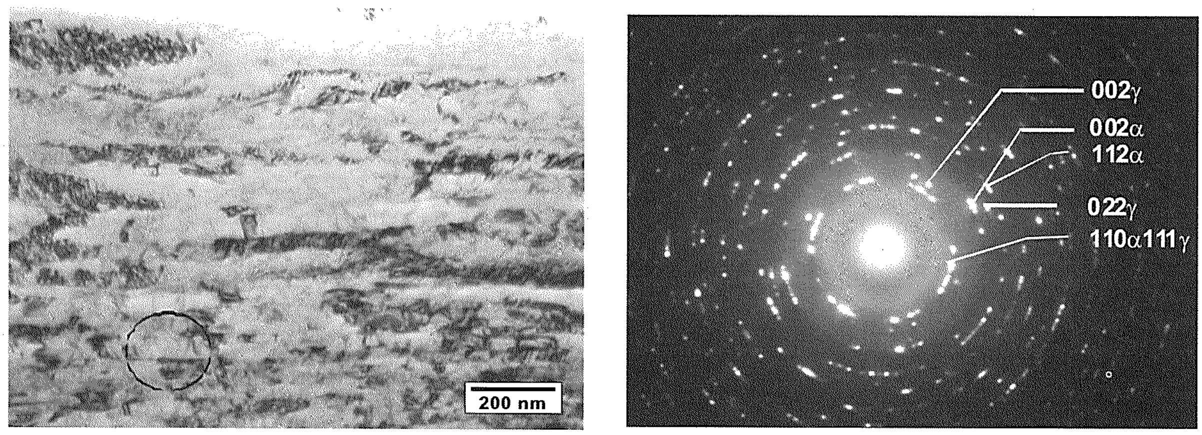

Fig. 6. TEM microstructure of the AISI 302 steel after 91.6% and annealing at 550°C for1 hour and SAED taken from the marked area

After annealing at 550°C and 650°C for 1 hour, the 111γ and 110α lines were separated and lines from austenite were sharpened. The results of X-ray phase analysis are presented in figure 1. With increasing annealing temperature, diffraction lines from martensite disappeared indicating at the occurrence of the reverse $\alpha'\rightarrow\gamma$ phase transformation (Fig. 1). The X-ray diagrams for the deformed and annealed wires exhibited changes in the intensities of diffraction line for both the γ- and α'-phases. The analysis revealed that at lower temperature only the reverse $\alpha'\rightarrow\gamma$ transformation occurred, whereas at higher temperature, i.e. 750°C and 850°C the process of austenite recrystallization took place in the structure additionally.

The changes of austenite and martensite texture upon annealing are presented in figures 2 and 3. After annealing of deformed material at 550°C the texture of autsenite became sharper and the intensity of martensite texture was reduced (Fig. 2). Such changes in texture intensity are due to the reverse $\alpha'\rightarrow\gamma$ transformation (Figs. 1÷3). The temperature increase up to 650°C led to the further reduction of the intensity of the martensite texture. Starting from the 750°C, the whole martensite was transformed and the recrystallisation process of the austenitic structure started up (Figs. 2, 3). A small decrease of the intensity of the main components of the austenite texture was observed but its fibre character was retained (Figs 2, 3). In the whole range of annealing temperatures the dominant component of the austenite texture was <111> orientation, accompared by the weaker <001> component. The maximum value of the ODF corresponded to the {110}<111> orientation. From the texture analysis and transformations experimental ODF (Figs. 2, 3) it results that the strain induced $\gamma\rightarrow\alpha'$ transformation and the reverse $\alpha'\rightarrow\gamma$ transformation proceeded according to Kurdjumov-Sachs (K-S) orientation relationship (Figs. 2, 3).

The obtained results corresponding with a number of other investigation concerning annealing of previously deformed metastable austenitic steels [14÷16]. It may be concluded that the reverse ($\alpha'\rightarrow\gamma$) transformation which presumably consisted of both, the shear and diffusional processes preceded the recrystallization which occurred at the temperatures 100-150°C higher.

4. Conclusions

1. When annealing the deformed wire made of metastable austenitic steel, two simultaneous processes occur, namely; the reverse $\alpha' \rightarrow \gamma$ phase transformation and the recrystallization of the austenitic γ-phase.
2. Base on the X-ray examination and microstructure analysis it is conducted that at the very beginning the reverse $\alpha' \rightarrow \gamma$ transformation occurs upon annealing. The transformation may proceed by sheer mechanism or diffusional process resulting in different dislocation density within the structure of austenite.
3. Following the reverse transformation the recrystallization processes take plays, that is the recrystallization of the deformed austenite and the austenite formed due to the ($\alpha' \rightarrow \gamma$) phase transformation.
4. During annealing the martensite texture is becoming weaker and finally martensite disappears due to $\alpha' \rightarrow \gamma$ transformation. The austenite annealing texture is the result of recrystallization of both the deformed austenite and the γ-phase formed due to the reverse transformation. The final texture consists of the major <111> component with some amount the <001> orientation.
5. In general it may be stated that in the case of metastable austenite the final microstructure after deformation and annealing is very fine. That is why it is concluded that the repeated processes of cold-working with subsequent annealing may lead to ultra-fine or even nano-crystalline structures displaying high strength and toughness.

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