Influence of $\gamma \rightarrow \alpha$ transformation temperature range bias on structural stability of laminar steel materials

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Abstracts. In this work laminar steel materials of W1-7 + AISI 304, W1-7 + AISI 430, 1008 + AISI 304 and 1008 + AISI 430 model compositions were studied. The $\gamma \rightarrow \alpha$ transformation temperature range bias and influence of such bias dynamics on the laminar structure stability was studied by the differential scanning calorimetry method. The study results have shown that only in the laminar steel material of W1-7 + AISI 304 model composition the $\gamma \rightarrow \alpha$ transformation temperature range bias to the low temperature region and the expansion of austenite field occurred, thus ensuring retention of the laminar structure in the process of material synthesis. Also, the nature of $\gamma \rightarrow \alpha$ transformation was analyzed for all four compositions which showed that the observed $\gamma \rightarrow \alpha$ transformation for each model composition of laminar steel materials was direct.

1. Introduction

At the present time the vigorous research activities are carried out in the mechanical engineering in the sphere of new classes of structural materials with better mechanical and operational properties than the traditional materials used at this point. A special class is distinguished among new classes of structural materials – laminar metal materials. Such materials possess the ultra-finely dispersed oriented structure. Thanks to such structure it is possible to implement a high set of mechanical and physical properties in the laminar metal materials, which will be much larger than that of the volume polycrystalline structural materials. This fact is demonstrated by the research in the sphere of thermal coefficient of linear expansion in laminar steel materials and thermal conductivity in laminar nanostructures [1,2].

The basis for laminar metal materials may be, for example, non-ferrous metals. The laminar metal materials based on non-ferrous metals are created by different methods of severe plastic deformation, e.g., accumulative roll bonding method (ARB method) or warm multi-pass caliber rolling method [3-6]. As a result, the materials can be created both on the homogeneous base, e.g., beryllium or copper base, and on the heterogeneous base, e.g., copper-nickel or niobium-copper composition base [7-10]. However, the final product, notwithstanding that it has the ultra-fine grain elongated structure, is a polycrystalline one and does not inherit laminarity. Besides, the use of the above mentioned methods of severe plastic deformation requires high time and energy expenditures.

The research of laminar metal materials based on ferrous metals - steels, i.e. the laminar steel materials is of greater interest to the mechanical engineering. Such materials are produced by hot packet rolling method being, which is more efficient in time and energy expenditures as compared to the severe plastic deformation methods [11]. Besides, a laminar structure is inherited in the laminar steel materials, consisting of hundreds (thousands) of layers with a micron (submicron) thickness. Creation of a laminar structure in the final product (Fig. 1a) makes it possible to obtain a higher set of mechanical (strength, impact, as well as fatigue) properties as compared to the traditional volume polycrystalline steel materials [11].
However, in the process of laminar steel materials production the important is not only creation of laminar structure in the materials but also its retention. Two key factors influence the laminar structure retention. Firstly, the process of deformation during the hot rolling of laminar steel materials must take place in the high-temperature austenite field (Fig. 2). I.e. the temperature of the laminar steel material slab $T_{\text{slab}}$ in the process of rolling must be higher than the critical point of $\gamma \rightarrow \alpha$ transformation $A_r1$. At that, in the process of laminar steel material production the slab thickness decrease takes place. Accordingly, the more the slab is thinned, the higher its actual temperature drop will be when entering the rolling mill rolls. For this reason, in order to maintain the necessary rolling temperature the additional heating is required.

**Figure 1.** Microstructure of the laminar steel material cross-section after the second technological cycle

**Figure 2.** Process diagram of laminar steel material rolling
Taking into consideration the mentioned above it can be said that in the process of laminar steel material production by hot rolling the following situation is possible. Beginning from a certain pass through the rolling mill, due to small slab thickness its actual temperature $T_{\text{slab}}$ will be lower than the critical point of $\gamma \rightarrow \alpha$ transformation $A_{\text{r1}}$. At that, application of multiple heating will lead to thermal cycling in the temperature range of $\gamma \rightarrow \alpha$ transformation. This, in its turn, will lead to multiple phase recrystallization, resulting in disturbance of laminarity of the structure being created (Fig. 1b). Disturbance of structure laminarity is unacceptable, since it leads to actual loss of a high set of mechanical properties.

Secondly, in the course of laminar metal materials synthesis the diffusion processes take place [12]. The research of alloying elements diffusion mobility in laminar steel materials in the process of rolling has shown that the interlayer diffusion of alloying elements leads to averaging of the chemical composition [13]. This, in its turn, leads to change in the critical points position of $\gamma \rightarrow \alpha$ transformation in the laminar steel material, i.e. $\gamma \rightarrow \alpha$ transformation temperature range bias.

Taking into account the above mentioned two factors and keeping in mind that the laminar steel material deformation process must be carried out in the high-temperature austenite region, the important task in the process of laminar steel materials research is investigation of the influence of $\gamma \rightarrow \alpha$ transformation temperature range bias on structural stability.

### 2. Materials and Research Methods

Taken as materials for the present research were laminar steel materials of four model compositions with a different degree of structuring and having different structural stability (Table 1).

| Model composition | Number of process cycle | Number of layers, pcs. | Stability of laminar structure |
|-------------------|-------------------------|------------------------|--------------------------------|
| W1-7 + AISI 304   | 1                       | 100                    | retained                       |
|                   | 2                       | ~2000                  |                                |
| W1-7 + AISI 430   | 1                       | 100                    | retained                       |
|                   | 2                       | ~2000                  | not retained                   |
| 1008 + AISI 304   | 1                       | 100                    | retained                       |
|                   | 2                       | ~2000                  | not retained                   |
| 1008 + AISI 430   | 1                       | 100                    | retained                       |
|                   | 2                       | ~2000                  | not retained                   |

In order to produce laminar steel materials the use was made of the developed experimental process route (Fig. 3) consisting of the technological cycles repeated one after another [11]. Two technological cycles were used for this research. At the beginning of the first technological cycle the sheets of two steel grades 0.5 mm thick were taken from which billets were cut out. The surface of billets was machined. Next, a laminar packet was formed from the billets, consisting of 100 alternating billets 50 of each steel grade. Then, a laminar packet was vacuumed and plastically deformed at a temperature of 1000 °C. Upon completion of the first technological cycle the laminar sheets 10 and 2 mm thick were produced (an individual layer thickness in the laminar sheet was 100 and 20 μm, respectively). These laminar sheets were used as an initial material for the second technological cycle consisting of similar operations. Upon completion of the second technological cycle the laminar sheets 10 and 2 mm thick were produced (an individual layer thickness in the laminar sheet was 5 and 1 μm, respectively).
Figure 3. Experimental process flow diagram of the laminar steel materials production [11]

Samples for research of $\gamma \rightarrow \alpha$ transformation temperature range were prepared from the produced laminar steel materials of four model compositions. For this purpose, the differential scanning calorimetry method was applied with the use of a calorimeter DSC 404 F1 Pegasus manufactured by Netzch company. Samples of the compositions under research were heated to 1000 °C in the inert argon medium, then held at this temperature for 5 minutes, after that cooled at a rate of 10 °C/min. As a result, the heating and cooling thermograms were obtained for each sample. However, only cooling thermograms are represented in this work, since $\gamma \rightarrow \alpha$ transformation nature was also analyzed for each model composition.

3. Results and discussion

The results of the $\gamma \rightarrow \alpha$ transformation temperature range research (Fig. 4) in the laminar steel material of model composition W1-7 + AISI 304 have shown that the $\gamma \rightarrow \alpha$ transformation start temperature in a sample with a layer thickness of 100 μm (laminar sheet thickness 10 mm; the first technological cycle) was $A_{\gamma 1} = 780$ °C, and the transformation end temperature was $A_{\gamma 2} = 715$ °C. The $\gamma \rightarrow \alpha$ transformation start temperature for a sample with a layer thickness of 20 μm (laminate sheet thickness 2 mm; the first technological cycle) was already $A_{\gamma 1} = 285$ °C, and the transformation end temperature was $A_{\gamma 2} = 255$ °C. For samples having passed the second technological cycle the $\gamma \rightarrow \alpha$ transformation start and end temperatures were in the low temperature region (not shown in Fig. 4). So, in this composition with the use of the second technological cycle the bias of the $\gamma \rightarrow \alpha$ transformation temperature range towards the low temperature region and extension of the austenite field occurred.
Prior to carrying out analysis of $\gamma \rightarrow \alpha$ transformation nature it is necessary to take into account the following fact. The research in the sphere of diffusion in the laminar steel materials has shown that equalization of the chemical composition of the laminar steel material layers takes place in the model composition W1-7 + AISI 304 [13]. I.e., upon completion of the second technological cycle the laminar steel material is synthetized with the averaged chemical composition of a layer: $\sim 0.4\%$ C, $\sim 9\%$ Cr and $\sim 5\%$ Ni. Due to absence of the continuous-cooling transformation diagram of austenite decay for steel with the similar chemical composition, the Schaeffler diagram was used for the $\gamma \rightarrow \alpha$ transformation character analysis [14]. According to this diagram and considering the averaged chemical composition of the laminar material layer of the model composition W1-7 + AISI 304, the nickel equivalent is $\text{Ni}_{\text{eq}} = 5 + 30 \times 0.4 = 17$. Chromium equivalent is $\text{Cr}_{\text{eq}} = 9$. According to the calculation of nickel and chromium equivalents the laminar steel material of this composition after the second technological cycle structurally relates to austenitic-martensitic class. So, the peaks visible on the cooling thermogram (Fig. 4) are indicative of a direct $\gamma \rightarrow \alpha$ transformation.

At the research of laminar steel material of other model composition W1-7 + AISI 430 a similar dynamic of $\gamma \rightarrow \alpha$ transformation temperature range bias was observed (Fig. 5). For a sample with a layer thickness of 100 µm (laminar sheet thickness 10 mm; the first technological cycle) the $\gamma \rightarrow \alpha$ transformation start temperature was $A_{\gamma 1} = 800\, ^{\circ}\text{C}$, and the transformation end temperature was $A_{\gamma 2} = 740\, ^{\circ}\text{C}$. For a sample with a layer thickness of 1 µm (laminar sheet thickness 2 mm; the second technological cycle) the $\gamma \rightarrow \alpha$ transformation start temperature was decreased to $A_{\gamma 1} = 385\, ^{\circ}\text{C}$, and the transformation end temperature was decreased to $A_{\gamma 2} = 325\, ^{\circ}\text{C}$. I.e., for such composition of the laminar steel material the $\gamma \rightarrow \alpha$ transformation temperature range bias took place towards the average temperature range.
Figure 5. Cooling thermogram of samples of laminar steel materials of model composition W1-7 + AISI 430

Considering the data of diffusion research in the laminar steel materials [13] it can be said that equalization of chemical composition of layers also takes place in the model composition W1-7 + AISI 430, and as a result of the second technological cycle the laminar steel material with the averaged chemical composition of a layer is synthesized: ~0.4 % C and ~9 % Cr. Using the continuous-cooling transformation diagram of austenite decay for steel Z40C14 having a similar chemical composition [15], with account of the cooling rate in the thermal-analytical experiment (10 °C/min) it can be said that the visible sharp peaks of γ→α transformation in Fig. 5 have a martensitic nature.

The research of γ→α transformation temperature range bias in the laminar steel material of a model composition 1008 + AISI 304 has shown the following results (Fig. 6). A sample with a layer thickness of 20 µm (laminar sheet thickness 2 mm; the first technological cycle) has a γ→α transformation start temperature $A_{\gamma_1} = 670$ °C and a transformation end temperature $A_{\alpha_2} = 515$ °C. The γ→α transformation start temperature in a sample with a layer thickness of 1 µm (laminar sheet thickness 2 mm; the second technological cycle) was $A_{\gamma_1} = 400$ °C, and the transformation end temperature was $A_{\alpha_2} = 305$ °C. So, the γ→α transformation temperature range bias occurred towards the average temperature region.
According to the data of diffusion research in the laminar steel material of model composition 1008 + AISI 304 [13] it was revealed that the chemical composition of layers in this composition is equalized and corresponds to the following average values: ~0.08 % C, ~9 % Cr and ~5 % Ni. Since the continuous-cooling transformation diagram of austenite decay for steel with a similar chemical composition is not available, then in order to analyse the $\gamma\rightarrow\alpha$ transformation nature the use can be made of the Schaeffler diagram [14]. Considering the chemical composition averaging in this model composition the nickel equivalent is $\text{Ni}_{\text{eq}} = 5 + 30 \times 0.08 = 7.4$. Chromium equivalent is $\text{Cr}_{\text{eq}} = 9$. According to these values of nickel and chromium equivalents the laminar steel material of this composition after the second technological cycle structurally relates to martensitic class. The sharp peaks visible on the cooling thermogram (Fig. 6) are indicative of the direct $\gamma\rightarrow\alpha$ transformation.

The research of the laminar steel material of the last model composition 1008 + AISI 430 has shown the following dynamics in the $\gamma\rightarrow\alpha$ transformation temperature range bias. The $\gamma\rightarrow\alpha$ transformation bias towards the average temperature region has occurred for a sample with a layer thickness 20 µm (the laminar sheet thickness 2 mm; the first technological cycle): the $\gamma\rightarrow\alpha$ transformation start temperature was $A_{\text{r1}} = 545 ^{\circ}\text{C}$, and transformation end temperature was $A_{\text{r2}} = 455 ^{\circ}\text{C}$ (Fig. 7).

**Figure 6.** Cooling thermogram of samples of laminar steel materials of model composition 1008+AISI 304

According to the data of diffusion research in the laminar steel material of model composition 1008 + AISI 304 [13] it was revealed that the chemical composition of layers in this composition is equalized and corresponds to the following average values: ~0.08 % C, ~9 % Cr and ~5 % Ni. Since the continuous-cooling transformation diagram of austenite decay for steel with a similar chemical composition is not available, then in order to analyse the $\gamma\rightarrow\alpha$ transformation nature the use can be made of the Schaeffler diagram [14]. Considering the chemical composition averaging in this model composition the nickel equivalent is $\text{Ni}_{\text{eq}} = 5 + 30 \times 0.08 = 7.4$. Chromium equivalent is $\text{Cr}_{\text{eq}} = 9$. According to these values of nickel and chromium equivalents the laminar steel material of this composition after the second technological cycle structurally relates to martensitic class. The sharp peaks visible on the cooling thermogram (Fig. 6) are indicative of the direct $\gamma\rightarrow\alpha$ transformation.

The research of the laminar steel material of the last model composition 1008 + AISI 430 has shown the following dynamics in the $\gamma\rightarrow\alpha$ transformation temperature range bias. The $\gamma\rightarrow\alpha$ transformation bias towards the average temperature region has occurred for a sample with a layer thickness 20 µm (the laminar sheet thickness 2 mm; the first technological cycle): the $\gamma\rightarrow\alpha$ transformation start temperature was $A_{\text{r1}} = 545 ^{\circ}\text{C}$, and transformation end temperature was $A_{\text{r2}} = 455 ^{\circ}\text{C}$ (Fig. 7).
Figure 7. Cooling thermogram of samples of laminar steel materials of model composition 1008 + AISI 430

Using the data of diffusion research in the laminar steel materials [13] it can be said that equalization of chemical composition of layers takes place in the model composition 1008 + AISI 430, and after the second technological cycle the laminar steel material has an averaged chemical composition of a layer: ~0.08 % C and ~9 % Cr. Using the continuous-cooling transformation diagram of austenite decay 13Cr (SAE 51405-51409) having a similar chemical composition [15], with account of the cooling rate in the thermal-analytical experiment (10 °C/min) it can be said that the visible sharp peaks of $\gamma \rightarrow \alpha$ transformation in Fig. 7 have a martensitic character.

4. Conclusion

The results of the research performed have shown that the $\gamma \rightarrow \alpha$ transformation bias and the expansion of austenite field occurred for all four model compositions of the laminar steel materials. However, if the $\gamma \rightarrow \alpha$ transformation temperature range in compositions W1-7 + AISI 430, 1008 + AISI 304 and 1008 + AISI 430 having passed the second technological cycle has biased towards the average temperature region, then the $\gamma \rightarrow \alpha$ transformation in the model composition W1-7 + AISI 304 has biased towards the low temperature region. The maximum expansion of the austenite field took place in the model composition W1-7 + AISI 304. Thanks to this, no thermal cycling in the range of $\gamma \rightarrow \alpha$ transformation during the laminar steel material synthesis took place in this composition, which ensured retaining of the structure laminarity after the second technological cycle. The analysis of the $\gamma \rightarrow \alpha$ transformation character for all four compositions has shown that the transformations observed during the experiment were the direct $\gamma \rightarrow \alpha$ transformations.

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