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On aging of high-strength stainless steel 08Cr15Ni5Cu2Ti

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Abstract. Based on our results and the results of the experimental studies previously conducted by other researchers, the analysis has been made of the influence of remelting methods on the character of the formation of the level of the impact strength KCU and its component KCV at aging of steel 08Cr15Ni5Cu2Ti prepared by two methods of remelting. The results of studying the influence of the remelting methods, heat treatment modes and impurity cleanness of steels on the level of KCU and the degree of its decrease at the temperatures of age hardening are presented. The factors causing the difference in the structure and phase composition in the steel formed by different remelting methods are established. The temperature range in which a significant decrease in KCU takes place has been determined. The hypothesis is offered about the role of interstitial impurities in the KCU decrease at aging.

1 Introduction

The desired set of properties of steels of this grade (high-strength Stainless) is attained by the selection of optimal conditions at quenching and aging in the temperature range of (400÷600)° C. The studied steel aging has been investigated in few works [1-3]; the matter was partially considered in monographs [4-9]. It is believed that the possible reasons for decreasing KCU at aging of chromium-nickel steels with the martensitic-austenitic structure are a small amount or absence of retained austenite, γ_{retain}, in the structure; coarse grain; the presence of embrittling phases on grain boundaries; the formation of segregations of harmful impurities and alloying elements, particularly, chromium, as the result of the separation process in the iron-chromium matrix [10], and their interaction with interstitial impurities [11-14].

The comparative study of the mechanical properties of steel 08Cr15Ni5Cu2Ti prepared by vacuum-arc and electroslag remelting (VAR and ESR) after quenching and aging shows that though the VAR-steel structure differs from that of ESR-steel in the grain size, larger tendency toward the grain growth at heating and small amount of γ_{retain}, the character of the formation of the mechanical properties is the same in both VAR- and ESR-steel [8]. However, the differece in structure, phase and chemical composition between remelting and even a smaller amount of gas impurities (Table 1) lead to the lower level of the VAR-steel mechanical properties. After aging for maximum strength, the impact strength appears to be the most unstable characteristics (inadequacy is 40% of the required level).

The investigation of the KCU formation in the studied steel prepared by the two remelting methods at aging and the determination of 'dangerous' temperature ranges with relation to the KCU decrease and factors influencing the position of the above temperature ranges is of interest.

2 Material and investigation techniques

The investigation was conducted on the industrial melts, the chemical compositions of which are given in Table 1. 1. For the investigation, samples from the steel prepared by the electroslag and vacuum-arc methods of remelting, ESR and VAR, were selected. The samples were heat-treated as follows: water quenching from the temperatures (950, 1000, 1050, 1200)°C. Aging was conducted in the temperature range of (300÷500)° C with the step of 25° C for 1 to 10 h. At quenching from 1000° C, the influence of the following preliminary conditions was studied: 1. Annealing 660° C, 6 h; 2. Annealing 660° C, 6 h × 2 times; 3. Annealing 520° C, 50 h; 4. Annealing 520° C, 50 h + 660° C, 6 h; 5. Annealing 750° C, 3 h + 520° C, 50 h + 660° C, 6h.
Before aging, the halves of the fractured samples after the impact strength test were checked for the presence of retained austenite (γ_{retained}) by the X-ray method. The impact strength (KCU) was determined according to GOSTs 9454-60 and 9455-60 (sample: type I); the crack

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Table 1. The chemical composition of steel 08Cr15Ni5Cu2Ti prepared by two methods of remelting

Remelt-ing methods	Content of element, %											
	C	Mn	S	P	Si	Cr	Ni	Cu	Ti	[O]	[N]	[H] $cm^3/100$ g
ESR	0.07	0.59	0.007	0.017	0.45	14.05	5.33	2.03	0.03	0.0034	0.0032	3.8
VAR	0.05	0.48	0.006	0.020	0.43	14.22	5.64	2.16	0.06	0.0017	0.011	1.67

development work (KCV) was determined according to B.A. Drozdovsky method on non-standard samples with the section 11×10 mm, notch depth 2 mm and fatigue crack 1 mm [15]. The degree of the impact strength (ΔKCU) decrease in the ESR- and VAR-steel was investigated in different conditions of quenching, and in dependence on the isothermal holding time in the temperature range of annealing in the two-phase (α+γ)-region conducted prior to quenching, and aging.

3 Results and discussion

The investigation of the impact strength of the ESR- and VAR-steel quenched from 1000° C after aging in the studied temperature range shows that in the quenched condition both melts have high values of KCU, 2.6 and 1.6 MJ/m^2, respectively (Fig. 1, *a*). The impact strength decrease starts from the temperature of 350° C; in ESR-steel it is 1.4 MJ/m^2 and in VAR-steel – 0.6 MJ/m^2 (half of that of ESR-steel) with the minimum at the temperatures (425÷450)° C. Such sharp impact strength variation is accompanied by a significant change in the fracture character. After aging at (300÷375)°C, from being completely ductile, the fracture becomes half-brittle at (425÷450)° C (the fiber fraction decreases to 40-50% in the fracture). After aging at (475÷500)° C, the impact strength increases to the previous or even higher values at purely ductile fracture. Starting with 375° C, γ_{retain} becomes unstable and its amount decreases from 6.5% in the ductile condition to 4.5% in the brittle condition.

The impact strength testing of the samples after quenching, cold treatment and aging show the similar dependence but with a greater decrease in KCU, which is 0.2 MJ/m^2 and 0.1 MJ/m^2 for ESR-steel and VAR-steel, respectively (Fig. 1, *b*). Thus, the impact strength decrease in the studied steel is not connected with the γ_{retain} decomposition since in the ductile condition, the steel without austenite has the same KCU values as the samples without cold treatment. The decrease of the retained austenite amount contributes into a general decrease in KCU; however, it is not a determining factor.

The aging temperature range, in which the KCU decrease takes place, shifts to the side of lower temperatures with the increasing duration of holding from 1 h to 10 h for both ESR- and VAR-steel. At holdings for 3h, 7h, and 10 h, the minimal impact strength values correspond to 425° C, and at holding for 1 h - to 450° C.

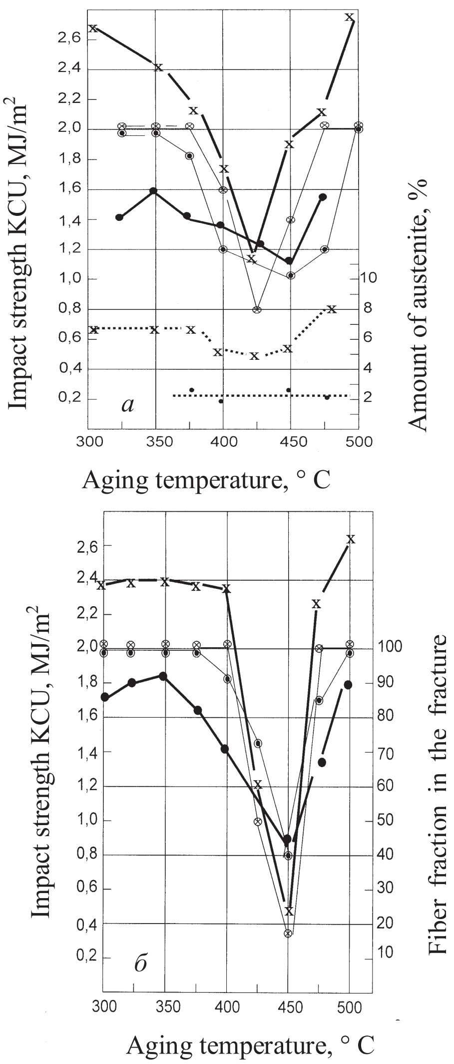


Fig. 1. The influence of the aging temperature on the impact strength (KCU), fiber fraction in the fracture and the amount of the retained austenite (γ) in steel 08Cr15Ni5Cu2Ti prepared by two remeltings methods.
a – after quenching from 1000° C;
b – *a* + cold-treatment at -70° C;
•- VAR, x – ESR – KCU and γ_{retain} (%);
• ©, ⊗ - ductile component (%) VAR and ESR, respectively. Aging for 3 h.

The degree of the impact strength decrease (ΔKCU) in the studied steels evaluated by the difference of KCU values in the ductile and brittle conditions (KCUduct - KCUbri) at holdings to 10 h in the ‘dangerous’ temperature range with relation to the KCU decrease is shown in Fig. 2. It can be seen that VAR-steel is much less tending to the loss of ductility than ESR-steel. The

ΔKCU value of VAR-steel increases steadily; in ESR-steel the processes leading to the impact strength decrease are taking place mainly for the first three hours (Fig. 2a, curve 2). At further holding to 10 h, the increase of ΔKCU is insignificant and it takes place due to the KCU decrease in the brittle condition. In addition, the change of the crack development work KCV at holding during aging also indicates the larger reliability of VAR-steel (Fig. 2b, curve 2). The results are confirmed by the shift of the transition temperature indicating the higher gas-and-interstitial-elements inclusion cleanness of VAR-steel [16]. However, in the VAR-melt with the high content of titanium required by OST (0.16%), the KCU decreases to the illegal values (Fig. 2c, curve 2).

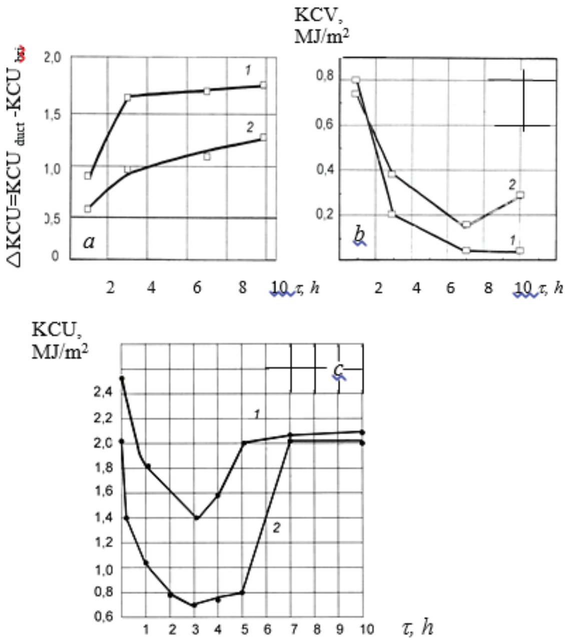
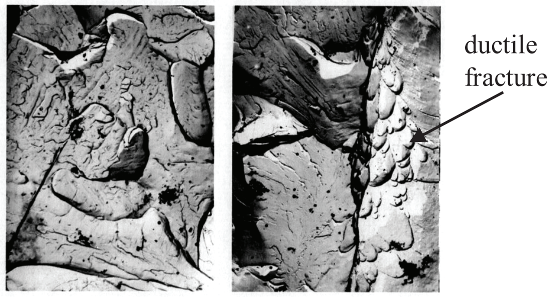


Fig. 2. The influence of the time of aging on the degree of the impact strength decrease (Δ KCU) and the work of the crack development (KCV) in the 'dangerous' temperature range. Steel 08Cr15Ni5Cu2Ti. Quenching temperature - 1000° C.
1 – ESR and 2 – VAR;
(*a*-2) and (*b*-2) - at Ti <0.06%; (*c*-2) - at Ti=0.16 %

On the electron micrograph of the sample with KCU = 0.28 MJ/m² (Fig. 3) it can be seen that the brittle fracture along the grain boundary has areas of ductile fracture (weakly elongated cups) situated along the retained austenite interlayers. Testing of cold-treated samples confirmed this fact. It can be suggested that the larger amount of austenite will make the KCU decrease smoother. The simultaneous presence of ductile and brittle components in the steel fracture indicates the competition of the two fracture mechanisms due to the strength balance of the boundaries and body of the grains strengthened by secondary phase precipitates. Unfortunately, the process of the ε-phase precipitation during aging has not been traced yet and it is impossible to link the temperature range of the ductility decrease with a certain degree of the dispersivity and distribution of the above phase.



Magnification: 3000 × 1.5

Fig. 3. Fractographs of the sample of ESR-steel 08Cr15Ni5Cu2Ti (KCU = 0.28 MJ/m²) after quenching from 1000° C and aging at 450° C, 2.5 h.

The degree and position of the KCU decrease interval are significantly influenced by the quenching temperature and the heat treatment conditions before quenching. If the studied steels quenching from 950-1050° C does not change the KCU-decrease temperature range, quenching from 1200° C makes the temperature range wider, especially for ESR-steel. The character of the influence of the quenching temperature and remelting method on ΔKCU is practically the same; both dependences have a minimum at 1000° C (Fig. 4). It means that at this temperature both remelts of the studied steel are less tending to the KCU decrease. In the range of 950° C - 1000° C the decrease of ΔKCU in ESR-steel is more intensive than that in VAR-steel. In addition, these dependences clearly show that independently of the austenization (quenching) temperature, at aging in the temperature range of (425÷450)° C, ESR-steel tends to the KCU decrease more significantly than VAR-steel at practically the same level of strength [8].

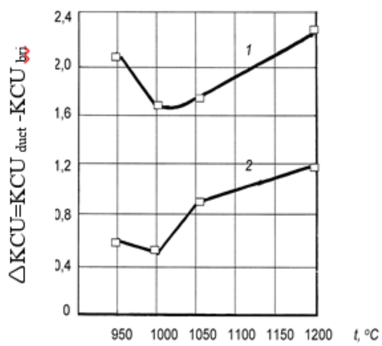


Fig. 4. The influence of the quenching temperature on the degree of the Δ KCU decrease in VAR - and ESR - steel 08Cr15Ni5Cu2Ti at aging for the strength maximum (425-450° C). 1 – ESR and 2 – VAR

The quenching temperature influences ΔKCU mainly via the grain size. At 1000-1050° C, the increase of ΔKCU in VAR-steel is more intensive than that in ESR-steel, and a noticeable growth of grain in VAR-steel starts at the temperature a little higher than 1000° C while in ESR-steel the grain growth starts at a much higher temperature (Fig. 5). However, the quenching temperature also influences the degree of ΔKCU with relation to obtaining the homogeneity of the solid solution at quenching. This is proved by the ΔKCU value

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which is higher after quenching from 950° C than that after quenching from 1000° C. At 950° C, the chromium (carbides, carbonitrides)-based complexes dissolve incompletely and even in the case of their complete dissolution, the homogeneity of the solid solution is not reached. It can be suggested that in the temperature range of (950÷1050)° C, the influence of two factors takes place, i.e. obtaining the more homogeneous solution leads to the KCU increase at aging in the temperature range of (425÷450)° C, and the grain size growth acts oppositely. At the temperatures higher than 1050° C, the grain growth makes a more significant contribution into ΔKCU.

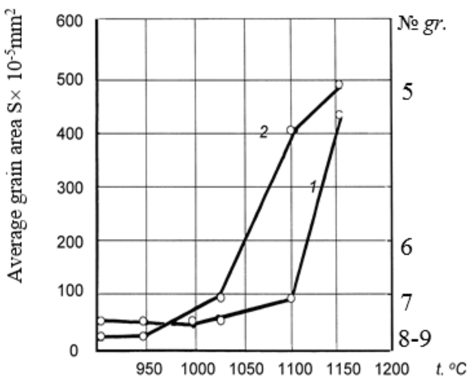

Fig. 5. The influence of the quenching temperature on the grain size in ESR-and VAR-steel 08Cr15Ni5Cu2Ti. 1- ESR, 2- VAR

In the heat treatment of the semifinished items from the studied steel, preliminary annealings in the two-phase (α+γ)-region are of special importance: one of them (750° C, 3 h + 520° C, 50 h) is conducted for removing diffusion-mobile hydrogen; the other (single or double annealing at 660° C, 6 h) stabilizes the mechanical properties. The investigation of the influence of annealings in the two-phase region conducted before quenching and followed by aging and of their combination show that annealings do not qualitatively change the character of the impact strength formation. The position of the 'dangerous' temperature range remains the same for VAR-steel (Fig. 6, *b*), and for ESR-steel it shifts by 25° C to the side of higher temperatures (Fig. 6, *a*). The stronger decrease of the ESR-steel impact strength is observed. However, in comparison with the samples after quenching, the annealings significantly increase the intensity of the KCU decrease after age-hardening: the most KCU decrease is at annealing (520° C, 50 h) at the VAR-steel aging – 0.7 MJ/m^2, and at the ESR-steel aging, annealing (750° C, 3 h + 520° C, 50 h + 660° C, 6 h) contributes into the KCU decrease - 0.8 mJ/m^2.

The question whether the influence of the processes taking place in the two-phase region still remains after heating for quenching is discussed in Ref. [13]. In the present work, the austenite condition obtained after double annealing and without annealing has been investigated by the method of internal friction and it is shown that the changes in the structure appearing at the stage of annealing in the (α+γ)-region remains after quenching due to the steadier and longer involvement of all the bulks of the metal into deformation in contrast to the avalanche-like beginning of the movement of dislocations in the metal without preliminary annealing.

At the same time, the influence of annealings on the tendency toward brittle fracture at decreasing temperature immediately after quenching is insignificant. The character of the serial curves is similar to that of the curves obtained after quenching from 1000° C without annealing: the curves of KCU = $f(T_{test})$ are flat for ESR- and VAR-steels without a distinct transition temperature, and the difference in the KCU level between the remelts remains [14]. Thus, the main contribution into the KCU decrease is made by the phase transformations at aging.

For understanding what causes the decrease of the impact strength and its component in the temperature range of (375÷450)° C and how the inclusion cleanness and chemical composition of the steel influence the impact strength, phase transformations at heating of hardened steel 08Cr15Ni5Cu2Ti have been analyzed. In the mentioned temperature range, in addition to the temper processes, precipitation of the disperse strengthening ε-phase rich in copper [5] and formation of concentration inhomogeneities in chromium [9,15], there are the processes of the redistribution of interstitial inclusions (carbon, nitrogen, oxygen) and harmful impurities as well. At heating temperatures (270÷320)° C, the setting of nitrogen, carbon and other atoms on dislocations with the formation of segregations takes place [13]. According to the results of the measurement of inner friction, the rise of the temperature to (380÷400)° C increases the coefficient of diffusion of the atoms and leads to the segregation dispersal. From the aging temperature of 320° C, the electrical resistance starts decreasing [2], and the impact strength also slightly decreases, and from the aging temperature of 350° C the impact strength decrease is more significant. Starting with 350° C the variation of the transition temperature is observed [14]: at the aging temperature of 375° C its shift is 40 deg. and at 400 °C – 60 deg.

Since the variation of the specific resistivity and the transition temperature shift indicate the separation processes characteristic of high-chromium ferrite, it is suggested that the separation is possible in the studied steel. The kinetic curve for the chromium regions formation in the studied temperature range of tempering and aging obtained by the NRG method [15] allows to conclude that at 450°C for 1 h, chromium-depleted and chromium-enriched regions are formed in the solid solution. In Ref. [16] it is stated that chromium enrichment of the regions is possible to 40%. At this temperature, the transition temperature shift in VAR- and ESR-steel is maximal [14]. However, there are no literature data that the separation significantly shifts the critical temperature of the transition of the steels of this grade to the brittle condition.

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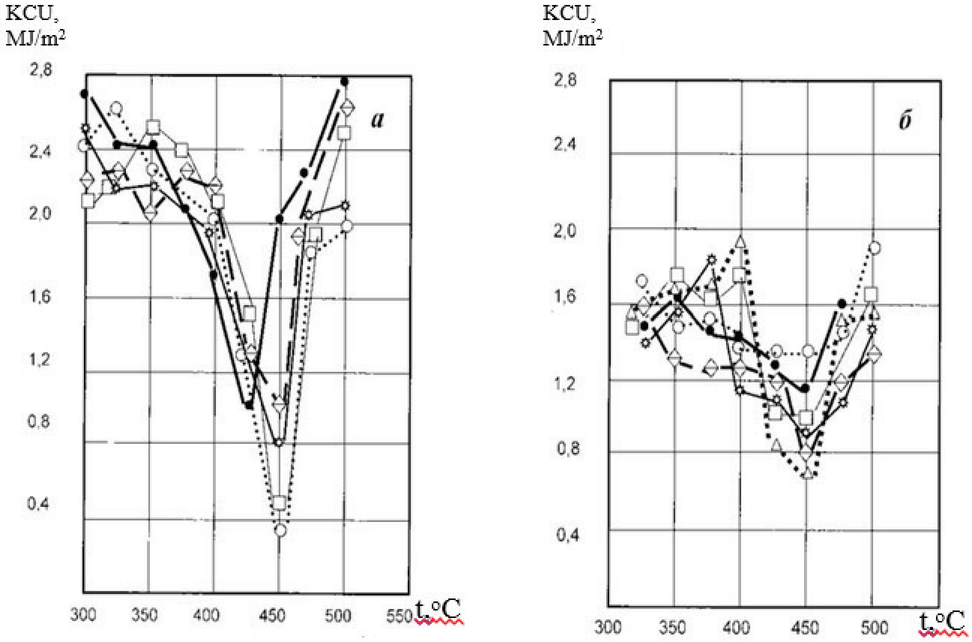

Fig. 6. The influence
and VAR (*b*) at differ

□ – 520° C, 50 h + 660° C, 6 h;
○ – 750° C, 3 h + 520° C, 50 h + 660° C, 6 h;
△ – 520° C, 50 h;
◇ – 660° C, 6 h. The time of aging is 3 h.

At the same time it is known [Problems of physics of strength, IFM Transactions ed. V.A. Arkharov] that interstitial impurities varying within 0.002 – 0.02% wt% can raise the transition temperature of pure chromium from (-100)° C to positive (above-zero) temperatures (~260)°C. The researchers in Ref. studying the influence of small amounts of impurities on the metal mechanical properties associate the transition of chromium from the ductile to brittle condition with the increase of the chromium resistance to plastic deformation and the decrease of σ_B under the influence of impurities, among which nitrogen produces the strongest influence on ductility. Thus, it can be suggested that the chromium-enriched regions can dissolve interstitial elements within the solubility limits at the studied temperature and contribute into the sharp manifestation of ΔKCU in the established intervals (Fig.1). The dimension of the appearing pure chromium regions is of importance; in the studied steel the dimension of theregions is ~5 nm according to X-ray and NGR data set.

The main hardening of the studied steel at aging takes place in the temperature range of (400÷450)° C according to the mechanism of dispersion hardening due to the precipitation of the disperse ε-phase based on copper and reaches (150÷200) MPa while the strength level in the hardened condition is up to (1150÷1200) MPa. By analogy with other steels and alloys, the corresponding decrease of the impact strength should be ~ (0.2÷0.4) MJ/m^2. In reality, the value of the KCU decrease does not correspond to the value of the martensite hardening due to the ε-phase precipitation. Thus, in addition to the influence of the ε-phase, the impact strength decrease happens due to another much stronger process. Possibly, this process is the formation of mixed regions consisting of chromium atoms and interstitial elements.

In addition, in all the experiments a very sharp transition from the low KCU values to the higher ones is noticed. It is sufficient to raise the aging temperature from 450 to 475° C for the impact strength to grow sharply to the values in the ductile condition. In this temperature range, the breakage of the coherency between the matrix and the strengthening ε-phase takes place. The increase of the temperature in the mentioned range at holding for 1 h cannot lead to the disappearance of the chromium-enriched and chromium-depleted regions and to the significant variation of the degree of the solid solution homogeneity. The ductility recovery most likely can be due to the decrease of the concentration of interstitial atoms in the chromium-enriched regions. At the dynamic equilibrium, the number of atoms getting to the chromium-enriched region is compensated by the number of atoms leaving the region.

Thus, the impact strength decrease in the studied temperature range can be divided into two stages. At the first stage (300÷400) °C the change of the properties can be explained by the martensitic matrix separation in chromium. At the second stage (400÷450)° C the loss of the matrix ductility takes place due to its hardening according to the dispersion hardening mechanism and further progress of the separation processes initiated by the precipitation of the disperse strengthening ε-phase. The chromium-enriched regions can be additionally 'embrittled' by interstitial elements in the amounts exceeding their equilibrium concentration.

The difference in the properties of the remelts of the studied steel is due to the difference in the short-range order structures [8] because of the non-equlibrium and inhomogeneity of the steel in the liquid state, gas-saturation, particularly, in interstitial elements, and high titanium content (Table 1).

The experiments show that in the aging process of steel 08Cr15Ni5Cu2Ti prepared by VAR- and ESR-methods, a significant impact strength decrease takes place. In practice, the KCU decrease remains unnoticeable due to the high value of the minimum KCU in a 'dangerous' temperature range. In the presence of retained austenite and fine grain, the impact strength level remains higher than the required one (0.8 MJ/m^2). In the case of coarse grain and small amount of austenite, the KCU decrease is very sharp as seen on the melt with high content of titanium (Fig.2, *curve* c).

4 Conclusions

1. The processes of the martensite decomposition in stainless steel 08Cr15Ni5Cu2Ti at heating in the tempering-and-aging temperature range of (270÷450) °C have been analyzed. In the mentioned range, in addition to the processes of tempering and precipitation of the disperse strengthening ε-phase, there are processes of the redistribution of interstitial elements (carbon, nitrogen, oxygen) and harmful impurities and the formation of the concentration inhomogeneities in chromium. Martensite of quenching decomposes into two solid solutions: the first is chromium-enriched and the second – chromium-depleted.
2. The temperature and time dependences of the variation of the impact strength KCU at aging of the studied ESR- and VAR-steel are established. In the aging temperature range of (375÷475)°C, the impact strength varies through the minimum at (425÷450) °C , the position of which depends on the remelting method, quenching temperature, modes of the preliminary treatment before quenching and time of holding in the 'dangerous' temperature range of aging (425÷450) °C.
   - The influence of the remelting method on the character of the KCU formation is expressed as the degree of the decrease of $\Delta KCU = KCU_{duc} - KCU_{bri}$ in the 'dangerous' temperature range of aging. This value is much lower in VAR-steel, in which the aging processes influencing ΔKCU are developing much slower and do not have clearly expressed character than those in ESR-steel.
   - The quenching temperature influences ΔKCU in both ESR- and VAR-steel due to the obtained homogeneity of the solid solution and the grain size.
   - The existing modes of the preliminary annealings before quenching of ESR- and VAR-steels intensify the KCU decrease after aging, but at the same time, the value of the minimum of the impact strength in the 'dangerous' temperature range increases.
   - The sharp impact strength decrease after aging is caused by the formation of mixed regions consisting of high-chromium martensite and interstitial atoms. Retained austenite plays the role of a background, on which the development of the processes leading to the KCU decrease takes place; therefore, there should be a sufficient amount of retained austenite in the steel structure.
3. It is shown that in the melt with a low content of titanium and gas impurities, despite the low level of γ_{retain} and KCU after quenching VAR-steel is more reliable after 1.5 h of aging for strength maximum.

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