

Processing of PK 324 Duplex Stainless Steel: Influence of aging temperature and cooling rates on precipitation - preliminary results

Preoblikovanje dupleksnega nerjavnega jekla PK 324: Vpliv temperature staranja in ohlajevalnih hitrosti na izločanje – preliminarni rezultati

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Abstract: Knowledge on precipitation phenomena in the PK 324 duplex stainless steel (DSS) is an important step in improving its final microstructure in the production process. Due to increased content of Cr, Ni and C the DSS exhibits complex microstructure, consisting of ferrite, austenite, intermetallic phases (σ) and carbides. Several tests were applied in order to obtain necessary knowledge for improving the processing of the DSS. Aging-treatment tests revealed that σ phase precipitated in the range between 680 °C and 900 °C. Additionally, the influence of cooling rates on the precipitation and transformation behaviour of samples, previously aged in the range between 950 °C and 1300 °C, has been studied. With sufficiently high cooling rates the precipitation of σ phase could be reduced to negligible values.

Izvleček: Razumevanje izločanja v dupleksnih nerjavnih jeklih PK 324 je pomemben korak k izboljšavi končne mikrostrukture v procesu izdelave. Zaradi povišane koncentracije Cr, Ni in C tvori ta vrsta jekel kompleksno mikrostrukturo, ki je sestavljena iz ferita, avstenita, intermetalnih spojin (σ -faza) in karbidov. Izvedenih je bilo več različnih preizkusov, ki so služili za nabiranje potrebnega znanja za izboljšanje procesov izdelovanja teh jekel. S staranjem je bilo ugotovljeno, da se σ -faza izloča v temperaturnem intervalu med 680 °C in 900 °C. Dodatno je bila narejena še analiza vpliva ohlajevalnih hitrosti na vzorce, ki so bili predhodno starani v temperaturnem intervalu med 950 °C in 1300 °C. Pokazalo se je, da se je mogoče izogniti izločanju σ -faze z zadostno visoko ohlajevalno hitrostjo.

Key words: PK 324 duplex stainless steel, aging treatment, cooling rates, precipitation

Ključne besede: dupleksno nerjavno jeklo PK 324, staranje, ohlajevalne hitrosti, precipitacija

INTRODUCTION

Duplex stainless steels, composed mainly of austenite and ferrite grains, possess excellent corrosion resistance as well as mechanical properties, i.e. good ductility and strength, where austenite contributes to the ductility while ferrite, which is harder, improves the strength and the welding characteristics. In the temperature range between 500 °C and 1100 °C the precipitation of secondary phases such as sigma phase (σ) - $(\text{FeNi})_x(\text{CrMo})_y$, intermetallic compound, chromium nitride (Cr_2N), secondary austenite (γ_2) and Chi phases (χ) takes place in this type of stainless steels. When the composition is changed in the nominal concentration region the range of precipitation usually changes. Larger content of the ferrite-stabilizing elements, such as Mo and Si promote the σ -phase precipitation during the cooling process, while on the other hand the austenite-stabilizing elements, such as Ni, Mn, C and N reduce the σ -phase precipitation. Type M_7C_3 and M_{23}C_6 carbides usually commence to precipitate at temperatures below 1100

°C or 950 °C, respectively. The precipitation takes place on grain boundaries between the ferrite and austenite grains as well as on other incoherent boundaries (non-metallic inclusions). The σ -phase is one of the most influential phases according to the Fe-Cr binary system. It usually precipitates in the temperature range between 600 °C and 1000 °C, is very brittle and consequently increases the hardness of stainless steel but decreases ductility, toughness, corrosion resistance and consequently cold and hot deformability. Cooling rate influences the amount of precipitated carbides as well as the amount of sigma and other phases and consequently the obtained mechanical properties. Thus an appropriate processing parameters should be chosen in order to obtain the desired properties of the stainless steel in question that are needed in further processing steps for its quality production. After hot forming, cold forming is usually applied during the manufacturing of items with fine dimensions (8 mm wire). The cold formability of stainless steel is highly influenced by present sigma phase as well

Table 1. Chemical compositions of various batches, used in our experiments

Tabela 1. Kemična sestava različnih šarž, uporabljenih pri preizkusih

BatchNo.		w(C)/%	w(Si)/%	w(Cr)/%	w(Mn)/%	w(Ni)/%	w(Mo)/%	w(S)/%	w(P)/%	w(Al)/%	w(Cu)/%
1	Φ 13	0.12	0.39	29.66	1.87	10.00	0.17	0.003	0.028	0.024	0.15
2	Φ 90	0.10	0.22	30.1	1.96	9.66	0.32	0.02	0.032	0.006	0.19
3	cast	0.10	0.26	30.1	1.91	9.5	0.04	0.004	0.020	0.007	0.06

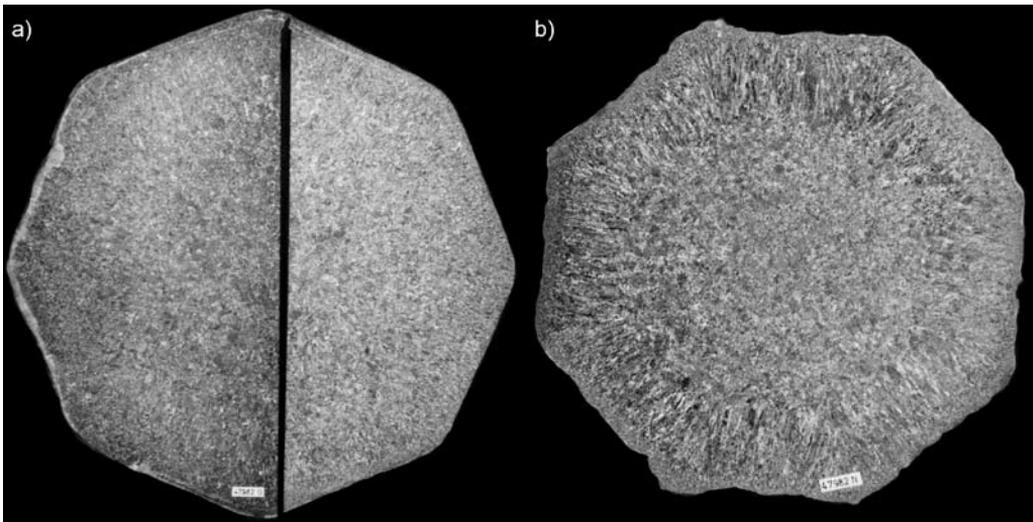


Figure 1. Observed macrostructures on the cross sections of the PK 324 DSS ingot; a) in the ingot head (G), and b) in the ingot foot (N)

Slika 1. Optična makrostruktura prečnega prereza ingota jekla PK 324; a) glava ingota (G) in b) noga ingota (N)

as by present carbides [1-14].

PK 324 is a ferrous alloy, usually used as welding material, grouped among super duplex stainless steel (DSS). Its production is still characterised by appearance of cracks as a consequence of precipitation processes. Thus this paper presents the results of influence of aging temperatures and cooling rates on the transformation and precipitation processes.

EXPERIMENTAL PROCEDURE

Applied materials, methods of characterization and analyses

The specimens made of PK 324 DSS and applied in testing were cut from a rolled wire with diameter $\Phi = 13$ mm (Batch no. 1), from a square rod (90 mm x 90 mm, Batch no. 2) and from 380 kg ingots prepared in a vacuum electric furnace (Batch

no. 3). Chemical compositions of the applied batches of the PK 324 DSS are given in Table 1. They differed from the standard DSS since they contained higher mass fraction of C (over 0.1 %), Cr (about 30 %), and Ni (about 10 %). An isopleths cross section of the Fe-Cr-Ni ternary diagram at 8 % Ni is given in [3].

(OM) ZEISS JENA VERT apparatus was applied for the light microscopy. Murakami's etchant (KLEMM [15]) was used to tint σ phase grey, α phase brown, carbides red, green and blue, while γ phase remained uncoloured. Further, XRD was used to verify the percentage of phases in the microstructure. Chemical compositions of the γ , α , σ phases and of carbides were determined with the EDS. Microanalyses were performed using the Leitz - AMR 1600 T scanning electron microscope and using the PGT IV EDX system.

Figure 1a and b present the macrostruc-

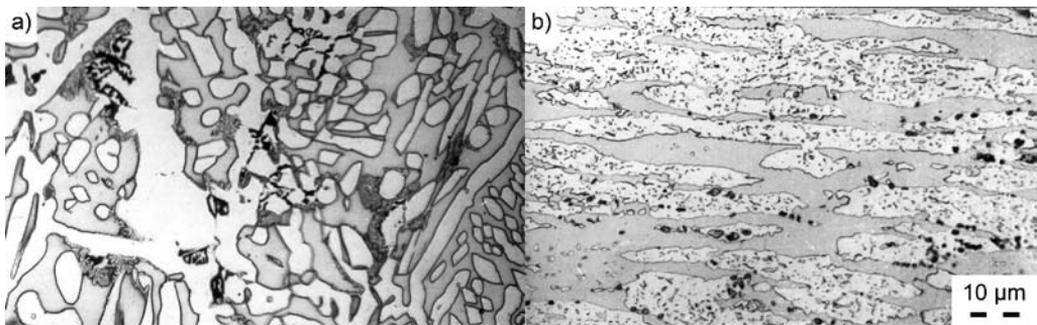


Figure 2. LM micrograph of PK 324 DSS; a) initial as-cast ingot; b) as-wrought, after 30 minutes of solution annealing at 1050 °C and subsequent water quenching
Slika 2. Posnetek mikrostrukture jekla PK 324; a) ulit ingot; b) predelan, po 30 min žarjenja na 1050 °C in po gašenju v vodi

tures in the cross-sections in the ingot's head (in the text marked with G) and in the ingot's foot (in the text marked with N), respectively. Figure 1a made evident that an area 5 mm below the surface of the ingot consists of globular grains. Towards the ingot centre, a 10 mm wide area consisting of dendritic grains can be observed while the areas further towards the centre contain equiaxial grains that gradually change into globular grains in the centre of ingot's head. The macrostructure of the cross section of ingot foot is presented in Figure 1b. The 5 mm wide area of globular grains in the outer part of ingot was clearly visible. Towards the ingot centre this area is followed by a 30 mm wide area of dendritic grains that gradually change into equiaxial grains and finally globular grains are found in the centre of ingot's foot.

The calculated value for $\text{Cr}_{\text{ekv}}/\text{Ni}_{\text{ekv}}$ ratio according to the equation given in [2] is 2.3, pointing to the presence of both phases at room temperature.

During the solidification process ferrite dendrites form first. After the solidification is complete the alloy consisted only of ferrite grains. Further cooling causes the α

$\rightarrow \gamma$ transformation and afterwards, due to the decreased solubility of C in austenite, precipitation of carbides takes place. With further cooling the decomposition of ferrite according to the $\alpha \rightarrow \sigma + \gamma$ eutectoid reaction takes place. During the cooling process the precipitation of carbides and of σ phase also takes place. Final microstructure consists of γ -phase in the interdendritic spaces, phases α and γ inside the dendrite grains, intermetallic phases (phases σ and χ) and eutectic carbides. Figure 2a shows the final as-cast microstructure of the batch no. 3 taken from the ingot centre. Type M_7C_3 and M_{23}C_6 carbides predominately precipitate on the α/γ boundaries in the interdendritic spaces (due to the segregation of C during the solidification process) and in a smaller extent also inside the grains close to the α/γ boundaries while σ -phase precipitates in the ferrite area. Measured volume fraction of ferrite in the ingot centre was about 48 %, austenite about 52 % and below 1 % of sigma phase and carbides at room temperature. In the samples that were taken from the ingot surface the volume fraction of ferrite was 45.2 %, of austenite about 54.8 % and below 1 % of

sigma phase and carbides.

In order to equalize fractions of ferrite and austenite in the initial microstructure, all the heat treated specimens were solution annealed in an electric furnace at 1050 °C for 30 min and quenched in water. According to the phase diagram given in reference [3] for this temperature approximately equal parts of α and γ were obtained. The obtained microstructure after aging heat treatment of the as-wrought material is presented in Figure 2b. The microstructure consists of approximately equal parts of ferrite and austenite and carbides (M_7C_3 and $M_{23}C_6$) which precipitated in austenite and on the α/γ interfaces. In austenite small islets of ferrite were found as a mixture of Widmannstätten microstructure and small islets of austenite.

High temperature aging treatments and cooling

After initial solution annealing the specimens underwent various aging treatments in the temperature range between 620 °C and 1050 °C in 20 °C increments. The tem-

perature was kept constant for 30 min. All the samples were quenched after the annealing process. Samples from batch no. 1 were applied for these tests. Based on these aging tests the temperature range of precipitation of sigma phase was determined. Detailed data of all the test conditions, characterizations, etc. that were performed in the research are given in Table 2.

To analyze the influence of cooling rates on the precipitation processes, the as-wrought (batch no. 2) and as-cast (batch no. 3) samples were quenched in water (with the cooling rate of approximately 200 °C/s) and cooled in air (with the cooling rate of approximately 4 °C/s), respectively, after they were aged for two hours in the temperature range between 1000 °C and 1300 °C. This served as a basis for characterisation of the influence of cooling rates (quenching in water, and cooling on air) on the achieved microstructure. Longer aging time (120 min) in the temperature range between 900 °C and 1250 °C (1300 °C) was chosen instead of shorter aging time (30 min) in the temperature range

Table 2. Applied aging conditions and applied tests

Tabela 2. Uporabljeni pogoji staranja in uporabljene preizkusi

	Temp. range 620–1050 °C	Temp. range 900–1250 (1300) °C	
Batch No.	1	2	3
Initial state	wrought, wire Φ 13 mm	wrought, Φ 90 mm	as-cast
Aging time (min)	30	120	120
Cooling	water	water, air	water, air
Microhardness: measured	Yes	No	No
OM, XRD, EDS	Yes	Yes	Yes

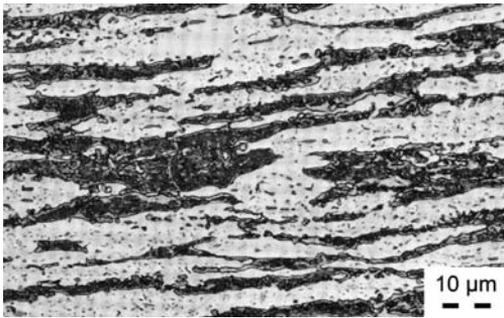
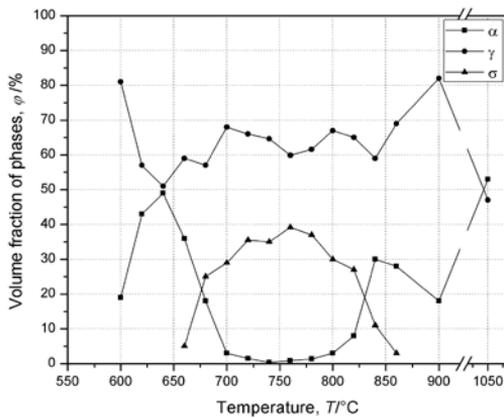


Figure 3. a) Volume concentrations of phases vs. aging temperature in the temperature range between 620 °C and 1050 °C, aging time of the as-wrought specimen ($\Phi = 13$ mm) was 30 min and b) the obtained LM microstructure of the as-wrought sample aged at 800°C for 30 min, (3% α , 30% σ , 67% γ)

Slika 3. a) Prostorninska koncentracija faz v odvisnosti od temperature staranja v območju med 620 °C in 1050 °C pri času staranja predelanega jekla ($\Phi = 13$ mm) 30 min; b) mikrostruktura, posneta z optičnim mikroskopom (3% α , 30% σ , 67% γ)

between 620 °C and 1050 °C, since the hot forming process usually takes place in that temperature range. This meant also longer holding times of workpiece at higher temperatures. Consequently the influence of cooling rate in the predetermined temperature range on the precipitation process was more relevant.

RESULTS

Stable microstructure, characterized by α/γ ratio, was obtained at 1050 °C and has become thermodynamically metastable at lower temperatures. This consequently results in the decomposition of ferrite phase into austenite and sigma phase ($\alpha \rightarrow \gamma + \sigma$). Figure 3a presents volumetric concentrations of phases vs. aging temperatures for aging time of 30 min in the temperature range between 620 °C and 1050°C. The samples were taken from batch no. 1. The sigma phase precipitated in the temperature interval between 660 °C and 860 °C, reaching maximum volume fraction of 39.5% at approximately 760 °C. Figure 3a makes evident that with the decomposition of α phase the amount of σ and γ phases increase. At the aging temperature of 1050 °C the obtained α/γ phase ratio was approximately 1 and indicates that it remained the same as after initial solution heat treatment. In Figure 3b the microstructure of the sample that was aged for 30 min at 800 °C is presented. The areas with ($\alpha \rightarrow \gamma + \sigma$) eutectoid are clearly visible; the sigma phase commenced to precipitate at the triple points as well as on the grain boundaries (α/α and α/γ) and it grew into the interior of ferrite grains.

The authenticity of the mentioned phases was also proved by micro-hardness measurements. The micro-hardness values $HV_{0.1}$ for ferrite (α) were in the range of 433–525, 230–309 for austenite (γ), austenite (γ) plus sigma phase (σ) in the range of 630–661 and of austenite (γ) plus carbide (K) in the range of 322–405. These values were in agreement with the obtained results by MARTINS et al [1, 4, 5].

As presented in Table 2, similar aging tests

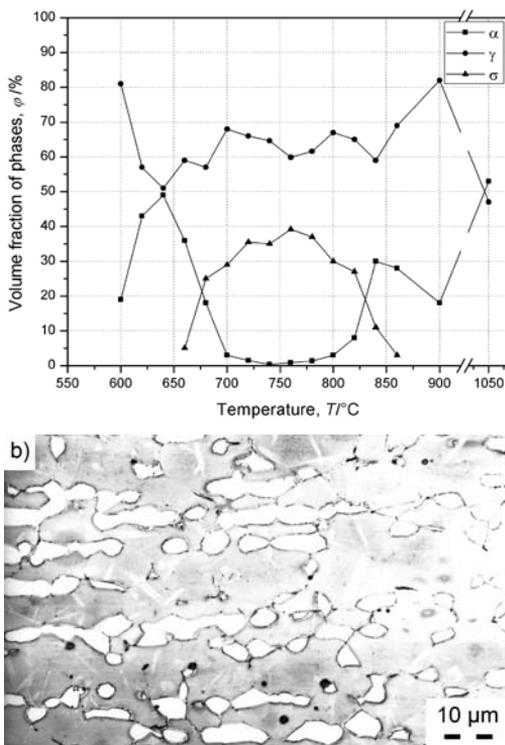


Figure 4. a) Volume concentration of phases after the two-hour aging vs. the aging temperature (900–1250 °C); b) microstructure of the specimen, aged at 1250 °C. As-wrought (Φ 90 mm), quenched in water, Batch no. 2.

Slika 4. a) Volumenska koncentracija faz po dveurnem staranju v odvisnosti od temperature staranja (900–1250 °C); b) mikrostruktura vzorca, staranega pri 1250 °C. Predelano jeklo (Φ 90 mm), gašeno v vodi, skupina 2.

for 120 min were carried out with specimens from the batches no. 2 and 3 in the temperature interval 900 °C and 1250 °C, being subsequently quenched in water and cooled in air in order to analyze the influence of cooling rates on the precipitation process. The volumetric concentration of phases, depending on the aging temperature, is presented in Figure 4a. It was evident that volume fraction about 3.5 % of

σ -phase appeared after 2 h aging at 900 °C. This could be ascribed to the increased content of Mo in the batch no. 2 ($w(\text{Mo}) = 0.32\%$) compared to batch no.1 ($w(\text{Mo}) = 0.17\%$). In general, the content of ferrite phase increased with the increasing temperature. Intensive $\gamma \rightarrow \alpha$ phase transformation commenced at the temperature around 1150 °C where at the temperature of 1250 °C the volume fraction of ferrite amounted to about 82 % but carbides were predominately dissolved (Figure 4b). On the other hand, the amount of ferrite in general decreased at temperatures below 1050 °C (down to 900 °C).

After all the specimens were aged for 2 h at selected temperatures in the range between 950 °C and 1300 °C they were cooled with two different cooling rates. They were quenched in water and cooled in air. The obtained microstructures of the as-cast and the as-wrought specimens are presented in Figures 5 and 6, respectively.

During the cooling of the as-cast specimens from the aging temperatures of 1300 °C and 1250 °C in water, the austenite and carbides precipitated predominately in ferrite and on the α/α grain boundaries (Figure 5a). At the lower cooling rate (i.e. in air) carbides precipitated predominately on the α/γ grain boundaries and to smaller extent in the ferrite phase (Figure 5b). Precipitated austenite had Widmannstätten morphology. The amount of precipitated carbides increased with decreasing the annealing temperature. The precipitation of carbides and austenite took place predominately on the α/γ phase grain boundaries (Figure 5c–d) and only a small difference was observed in the obtained microstructures of specimens that were quenched in water and cooled on air.

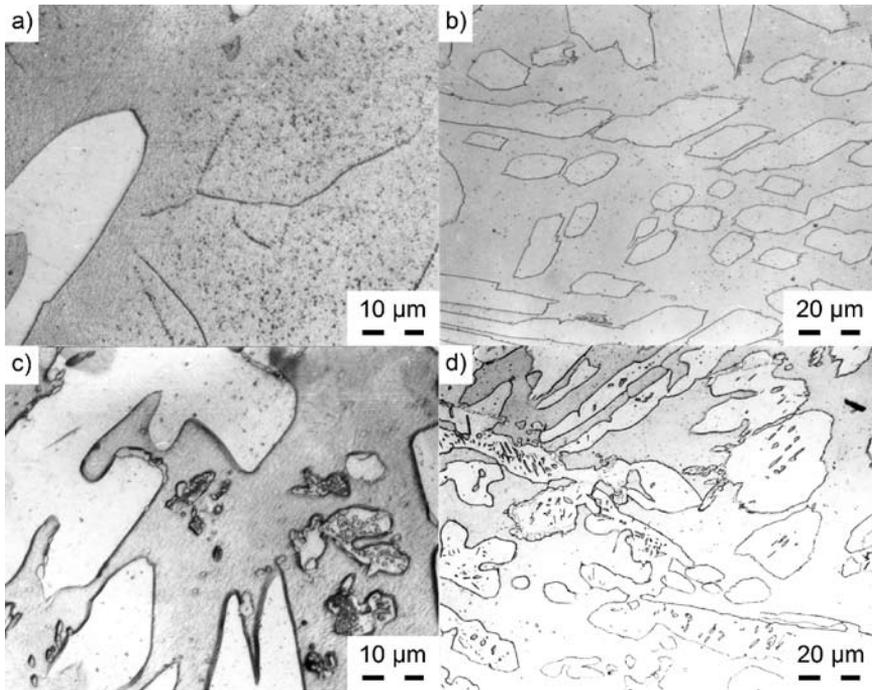


Figure 5. Obtained microstructures of the as-cast specimens of the batch no. 3 that were aged for 2 h at 1250 °C and consequently quenched in water (a) and air cooled (b); aging at 1050 °C, quenched in water (c), and air cooled (d), LM
Slika 5. Mikrostruktura, dobljena z optičnim mikroskopom vzorcev litega stanja, skupina 3, starana 2 h pri temperaturi 1250 °C in gašena v vodi (a) ter ohlajana na zraku (b); staranje pri temperaturi 1050 °C gašena v vodi (c) in ohlajana na zraku (d)

Similar results were obtained for the as-wrought specimens, where the only difference was that carbides precipitated during the quenching in water from the annealing temperature of 1300 °C and 1250 °C to smaller extent in the ferrite phase and to greater extent on the α/γ grain boundaries (Figures 6a-b, compared to Figures 5a-b). The annealing temperature and the cooling rate influenced the morphology of the precipitated carbides in the as-cast as well as in the as-wrought specimens. At higher annealing temperature the morphology of precipitated carbides was lamellar but with decrease of annealing temperature the mentioned mor-

phology slightly changed from lamellar to spherical. This change occurred in the as-wrought specimens that were quenched in water from the temperature range between 1150 °C and 1100 °C (Figure 6d) while in the specimens that were cooled in air this phenomenon occurred already at approximately 1175 °C (Figure 6c).

With the decrease of aging temperature (1050 °C) the amount of precipitated carbides increased. They predominately precipitated on the α/γ grain boundaries and their form varied from stick-shaped to spherical. The morphology of precipitates did not change only with the aging

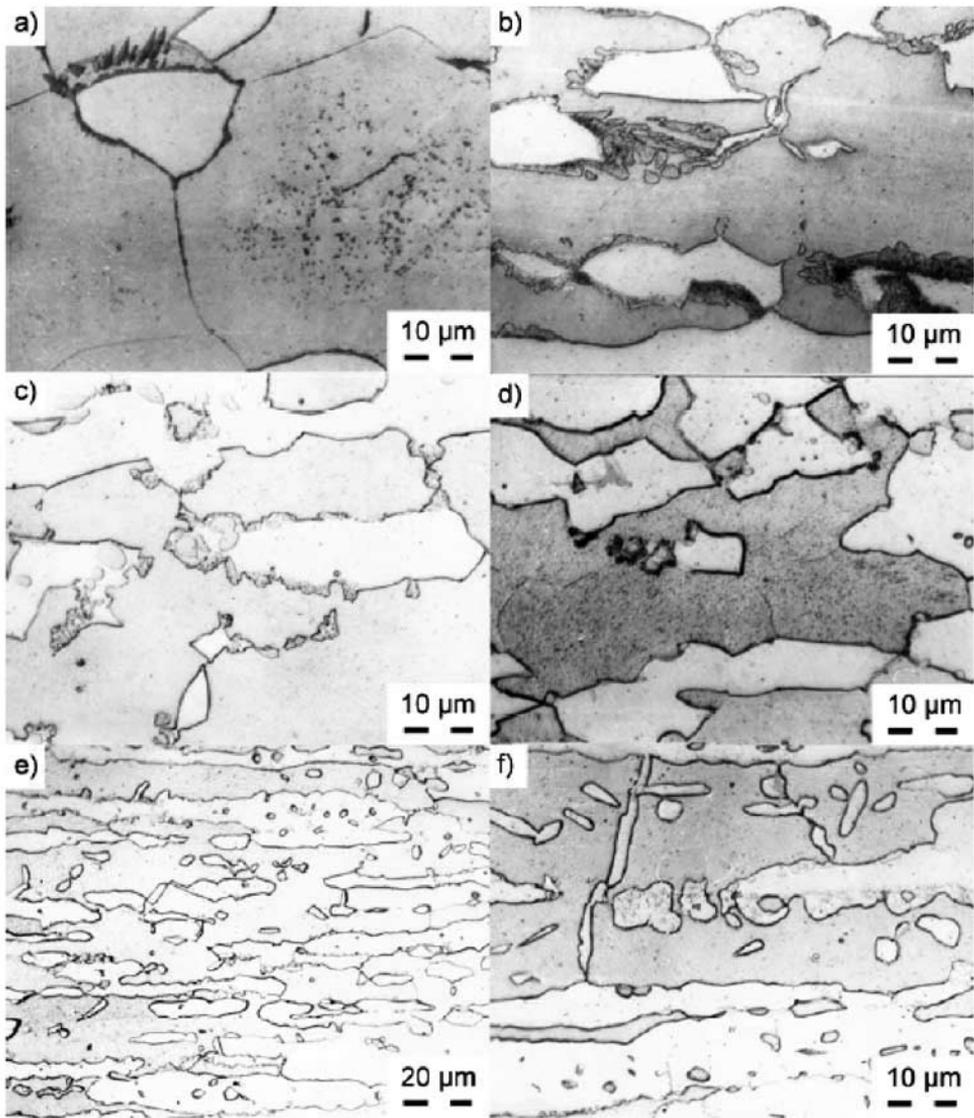


Figure 6. Obtained microstructures of the as-wrought specimens from the batch no. 2 being aged for 2 h in the temperature range of 1300–1000 °C, and subsequently quenched in water or cooled in air; aging at 1250 °C, water quenching (a), aging at 1250 °C, air cooling (b), aging at 1175 °C, air cooling (c), aging at 1150 °C, water quenching (d), aging at 1050 °C, water quenching (e) and air cooling (f), LM

Slika 6. Mikrostruktura, dobljena z optičnim mikroskopom predelanega jekla, skupina 2, po dvournem staranju pri temperaturi 1250 °C, gašena v vodi (a) ter ohlajana na zraku (b); staranje pri temperaturi 1050 °C, gašena v vodi (c) in ohlajana na zraku (d); staranje pri 1050 °C in ohlajana v vodi (e) ter na zraku (f)

temperature but also with the cooling rate (Figures 6e–f).

Examination with the EDS revealed that $M_{23}C_6$ -type carbides precipitated on the α/γ grain boundaries.

Table 3. Chemical composition (w/%) of phases, and the ($I\alpha/\gamma$) distribution coefficient of elements in the sample aged for 30 min at 1050 °C, batch no. 1

Tabela 3. Kemična sestava (w/%) faz in razdelitveni koeficient ($I\alpha/\gamma$) faz v vzorcih, starih 30 min pri temperaturi 1050 °C, skupina 1.

w/%	α	γ	Carbides	$I\alpha/\gamma$
Cr	33.76	25.75	68.12	1.32
Mn	1.98	2.31	1.57	0.86
Fe	55.78	58.71	20.17	0.95
Co	0.35	0.45	0.11	0.78
Ni	7.07	12.68	2.69	0.56
Mo	0.22	0.13	0.61	1.69

The amounts of α and γ -phases depend on the annealing temperature where as the cooling rate showed a negligible influence. Chemical analyses of austenite, ferrite and carbides (M_7C_3) in the specimen that was aged for 30 min at 1050 °C were performed using the EDS microanalyzer. They are presented in Table 3 together with the ($I\alpha/\gamma$) distribution coefficient. The highest difference of chemical elements content between ferrite and austenite was found for Mo and Ni, followed by Cr, Co, Mn and Fe. Transformation of ferrite into austenite took place only by diffusion of Cr and Mo to certain regions (carbides) thus enriching them and leading to the formation of sigma phase. The regions that were enriched with the mentioned elements due to segregation were favourable for commencement of the $\alpha \rightarrow \gamma$ transformation [6]. The carbides (M_7C_3) were mainly composed of Cr and Fe (see Table 3). The presence of M_7C_3 carbides

was discovered on the base of transforming the chemical composition of carbides from mass fractions into molar fractions, taking in account that molar fraction of carbon, N_C , was obtained by subtracting the sum of molar fractions of the other alloying elements that were composing carbides from the value 1. The calculated ratio between the sum of molar fractions of chromium and iron ($x(\text{Cr}) + x(\text{Fe})$) and the $x(\text{C})$ was approximately 7 : 3 that indicated the presence of M_7C_3 -type carbides.

CONCLUSIONS

Precipitation behaviour in the PK 324 ferrous alloy at various testing conditions was examined. Two aging treatment tests were applied; shorter aging time (30 min) in lower temperature range (620–900 °C (1050 °C)), and longer aging time (120 min) in higher temperature range (900–1300 °C). The aged specimens in the higher temperature range were cooled with two cooling rates; i.e. quenched in water and cooled in air. The following conclusions, useful for increasing the final quality of PK 324 ferrous alloy, can be stated:

- As-cast microstructure consisted of austenite, ferrite, sigma phase and M_7C_3 and $M_{23}C_6$ carbides. The carbides were mainly composed of Cr and Fe.
- The sigma phase nucleated on ferrite-austenite interfaces and grew into ferrite grains.
- The sigma phase precipitated in the temperature range of 660–860 °C, having peak value of the maximal amount of 39.5 % at about 750 °C and it was then completely dissolved at about 900 °C. The upper temperature limit of the σ phase precipitation is depended on the chemical

composition. The final hot forming steps should be performed above 900 °C.

- The concentration of sigma phase could be minimized if the cooling rate in the range of its precipitation was high enough. This would improve the cold deformability as well as the final microstructure.
- The 2 h aging process at about 1250 °C, gave the content of ferrite above 80 %; at around 1100 °C this content was reduced below 60 %, at around 1000 °C the content was already below 50 %, and at around 900 °C the content of ferrite was even lower than 40 %.

POVZETEK

Raziskana je bila precipitacija v dupleksnem jeklu PK 324 pri različnih pogojih preizkušanja. Uporabljena sta bila dva poteka preizkušanja: kratki preizkus staranja (30 min) pri nižjih temperaturah (620–900 °C (1050 °C)) ter daljši preizkusi staranja (120 min) pri višjih temperaturah (900–1300 °C). Preizkušanci so bili ohlajani na sobno temperaturo z dvema različnima hitrostma ohlajanja; gašeni so bili v vodi, drugi pa so bili ohlajani na zraku.

Pri tem so bile dobljene naslednje ugotovitve:

- Lita struktura je vsebovala avstenit, ferit, fazo sigma ter karbida M_7C_3 in $M_{23}C_6$. Karbida sta v večji meri vsebovala Cr in Fe.
- Faza sigma je nukleirala na fazni meji avstenit/ferit in je rastla v notranjost feritnih zrn.
- Faza sigma se je izločala v temperaturnem območju med 660 °C ter 860 °C, vrh ($\varphi = 39.5\%$) pa je dosegla pri 750 °C. Popolnoma je bila raztopljena pri

900 °C. Ta temperatura je odvisna od kemijske sestave in kaže na to, da bi se morali zadnji vtiki med kovanjem izvesti nad 900 °C.

- Koncentracijo izločene faze sigma bi bilo mogoče zmanjšati s pospešenim ohlajanjem skozi področje njenega izločanja. S tem bi se izboljšala preoblikovalnost v hladnem ter končna mikrostruktura izdelka.
- Dveurni proces staranja pri 1250 °C je pokazal, da je v mikrostrukturi prostorski delež ferita večji od 80 %. Pri 1100 °C je bilo ferita le še 60 %, pri 1000 °C 50 % ter pri 900 °C manj kot 40 %.

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