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Role of Cr and P additions in the development of microstructure and texture in annealed low carbon steels

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Abstract

The recrystallisation behaviour of four warm rolled steels was investigated during annealing. The extra-low carbon (ELC) steel displayed the highest rate of recrystallisation, the steels with additions of chromium and phosphorus (LC(Cr)), (LC(Cr,P)) recrystallised at intermediate rates, while the interstitial-free (IF) steel exhibited the lowest rate. The additions of Cr and Cr/P increased the fraction of g-fibre in the annealing textures compared to that present in the ELC steel; this effect was particularly pronounced up to 50 % recrystallisation. After the completion of recrystallisation, the steel textures were characterized by a dominant g-fibre in the IF steel, while in the three LC steels, the RD fibre was the principal one.

Keywords

carbon, p, role, low, microstructure, texture, steels, cr, additions, annealed, development

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ROLE OF Cr AND P ADDITIONS IN THE DEVELOPMENT OF MICROSTRUCTURE AND TEXTURE IN ANNEALED LOW CARBON STEELS

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The recrystallisation behaviour of four warm rolled steels was investigated during annealing. The extra-low carbon (ELC) steel displayed the highest rate of recrystallisation, the steels with additions of chromium and phosphorus (LC(Cr)), (LC(Cr,P)) recrystallised at intermediate rates, while the interstitial-free (IF) steel exhibited the lowest rate. The additions of Cr and Cr/P increased the fraction of γ-fibre in the annealing textures compared to that present in the ELC steel; this effect was particularly pronounced up to 50 % recrystallisation. After the completion of recrystallisation, the steel textures were characterized by a dominant γ-fibre in the IF steel, while in the three LC steels, the RD fibre was the principal one.

Key words: *low carbon steel, Cr and P additions, annealing texture, shear bands*

Uloga dodataka Cr i P u razvoju mikrostrukture i teksture u žarenim niskugljičnim čelicima. Ispitivana je rekristalizacija četiri tipa toplo valjanih čelika tijekom žarenja. Čelik s ekstra niskim ugljikom (ELC) pokazao je najveću brzinu rekristalizacije, čelici s dodatkom kroma i fosfora (LC(Cr)), (LC(Cr,P)) pokazali su srednje brzine rekristalizacije, dok je intersticijski-slobodan čelik (IF) imao najmanju brzinu. Dodavanje Cr i Cr/P povećalo je frakciju γ-vlaknastog materijala u žarenim teksturama u usporedbi s onom koju susrećemo u ELC čeliku; taj efekt je posebno izražen u 50 %-postotnoj rekristalizaciji. Nakon završetka rekristalizacije, teksture čelika su obilježene dominacijom γ-vlaknastog tkiva u IF-čeliku, dok je u tri LC čelika RD-vlakno bilo glavno.

Ključne riječi: *niskougljični čelik, dodaci Cr i P, žarena tekstura, smične trake*

INTRODUCTION

High mean *r*-value, which indicates good formability, is one of the necessary properties of steel sheet products designated for deep drawing applications. It has been known for some time that such high *r*-values require the presence of a strong {111} texture component lying in the rolling plane (i.e. the γ-fibre) [1]. However, intense <111>//ND fibres are generally only observed in warm rolled and annealed *interstitial-free* steels, while warm rolled and annealed low carbon steels exhibit poor formability and weak γ-fibres [2].

The development of the γ-fibre in annealed steels has been linked [2, 3] to the presence of a high volume fraction of grains containing shear bands after warm rolling. These bands appear to be the nucleation sites for recrystallised grains of the desirable orientation. After warm rolling, many of the grains in interstitial-free (IF) steels contain such shear bands and the steels then exhibit good forming characteristics after annealing [4]. By contrast, in low carbon steels, the presence of carbon in solid solution leads to dynamic strain aging (DSA) during warm rolling and to high positive rate sensitivities. The latter prevent the formation of high densities of in-grain shear bands, leading to a lack of nucleation sites for the γ-fibre during annealing.

One of the approaches towards improving the annealing texture in warm rolled low carbon steels is to remove carbon from solid solution, thereby reducing the extent of DSA and lowering the rate sensitivity of the flow stress. This stratagem is expected to encourage the formation of grains containing in-grain shear bands during warm rolling [5], an outcome that can be encouraged by alloying with carbide forming elements such as chromium. However, it has been shown that to form a strong ND fibre texture component and to improve the formability, the addition of a relatively high amount of Cr (1,3 wt %) is required [6]. The possibility of cost reduction by lowering

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the level of chromium in conjunction with boron or phosphorus addition has been studied in [7 - 9]. In the current work, the kinetics of recrystallisation and texture development at 700 °C have been followed in four warm rolled low carbon steels with and without additions of Cr and P. In particular, the main focus was on characterising the nucleation sites for the {111} fibre grains.

EXPERIMENTAL PROCEDURE

The chemistries of the materials used are given in Table 1. Two regular production alloys, an unalloyed extra low carbon steel (ELC) and an interstitial-free (IF) steel stabilized with titanium, were used. Two experimental chromium- (LC(Cr)) and phosphorus-modified (LC(Cr,P)) low carbon steels were obtained from Dofasco Inc., Hamilton, Canada. Warm rolling was conducted at 640 °C and average strain rates of 30 s^{-1} to 65 % reduction in a single pass on the EMR-CANMET pilot mill in Ottawa, Canada. Specimens for analysis were cut from the mid-width sections of the as-rolled strips perpendicular to the transverse direction. Annealing of the slices, covered with alumina powder to reduce oxidation and decarburisation, was carried out in an open atmosphere furnace at 700 °C. The annealing time was varied from 10 s to 2000 s to attain recrystallisation nucleus volume fractions of 5 %, 15 %, 50 % and 100 %.

Table 1. **Steel Compositions / wt. %**
Tablica 1. **Sastavi čelika / tež. %**

Steel	C	Mn	P	Cr	Ti	Al	N
IF	0.004	0.15	0.005	0.065	0.062	0.041	0.0037
ELC	0.020	0.12	0.004	0.071	-	0.048	0.0067
LC(Cr)	0.037	0.35	-	0.48	-	0.036	0.0012
LC(Cr, P)	0.039	0.32	0.04	0.04	-	0.028	0.0024

Samples for optical and scanning electron microscopy (SEM) were electropolished in 10 % perchloric acid, after which they were etched in 0,2 % nital for 10 min. The SEM study was conducted using a JEOL JSM-840 microscope in association with an Oxford Instruments EBSD system and a LEO 1530 FEG microscope. HKL Channel 5 and VMAP software were used for analysis of the EBSD data and measurement of the average recrystallised grain size. A rotation angle of 20° was employed to determine the grain orientations present. The orientation distribution functions (ODF's) were calculated from the measured pole figure data and are presented using the φ_2= 45° sections of Bunge's Euler space [9]. The recrystallised grains were deemed to be $\geq$ 1 μm in size with misorientations less than 2° within them, as based on the EBSD analysis. The volume fractions of recrystallised grains were determined from the EBSD data.

Table 2. **Observations on the nucleation of recrystallised grains**
Tablica 2. **Promatranja stvaranja nukleusa rekristaliziranih zrna**

Steel	Volume Fraction of Recrystallised Nuclei / %	Nucleation at Shear Bands		Nucleation at Grain Boundaries		Nucleation within Pearlite Colonies	
		Fraction / %	Average size / µm	Fraction / %	Average size / µm	Fraction / %	Average size / µm
IF	5	90	1.8 ± 0.7	10	8.0 ± 2.8	-	-
	15	68	4.4 ± 1.7	32	11.3 ± 2.1	-	-
ELC	5	57	1.5 ± 0.5	43	6.5 ± 1.4	-	-
	15	31	4.9 ± 1.8	60	8.5 ± 2.1	9	5.2 ± 1.4
LC(Cr)	5	68	1.9 ± 0.5	32	7.0 ± 2.1	-	-
	15	44	4.8 ± 1.4	45	11.4 ± 2.8	11	5.7 ± 2.6
LC(Cr,P)	5	55	1.7 ± 0.3	45	6.0 ± 1.2	-	-
	15	30	6.1 ± 1.9	54	10.4 ± 2.4	16	5.9 ± 2.7

Transmission electron microscopy (TEM) investigation was performed using a Philips CM20 microscope operated at 200 kV. Thin foils were produced by twin jet electropolishing using a solution of 5 % perchloric acid in methanol at – 30 °C and an operating voltage of 50 V.

RESULTS

The recrystallisation kinetics of all the steels studied are shown in Figure 1. The ELC steel recrystallised at a rate faster than all the others. The IF steel displayed the slowest recrystallisation rate. The LC(Cr) and LC(Cr,P) steels had similar recrystallisation behaviours and intermediate rates of recrystallisation (Figure 1.).

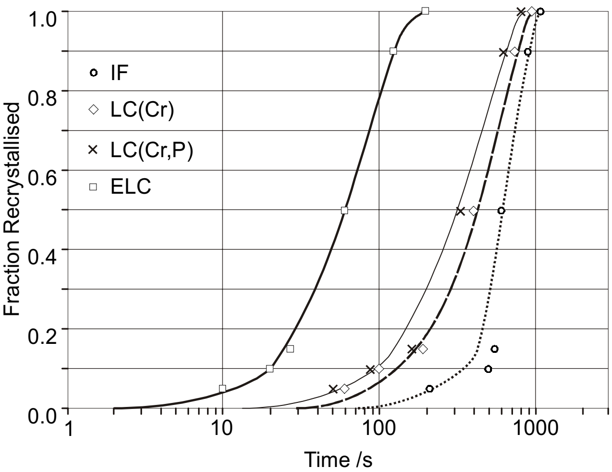

Figure 1. Recrystallisation curves for the IF, ELC, LC(Cr) and LC(Cr,P) steels warm rolled at 640 °C and annealed at 700 °C
Slika 1. Krivulje rekristalizacije za čelike IF, ELC, LC(Cr) i LC(Cr,P) toplo valjane na 640 °C i žarene na 700 °C

Both the TEM and SEM studies revealed that new strain-free grains with high degrees of misorientation with respect to the deformed matrix formed at three different locations within the microstructure: i) grain boundaries, ii) shear bands, and iii) pearlite colonies (Figures 2. and 3. and Table 2.). Nevertheless, the preferred nucleation sites were the shear bands and the IF and LC(Cr) steels displayed higher such volume fractions during the early stages of recrystallisation (volume fraction < 15 %) than the ELC and LC(Cr,P) steels (Table 2.).

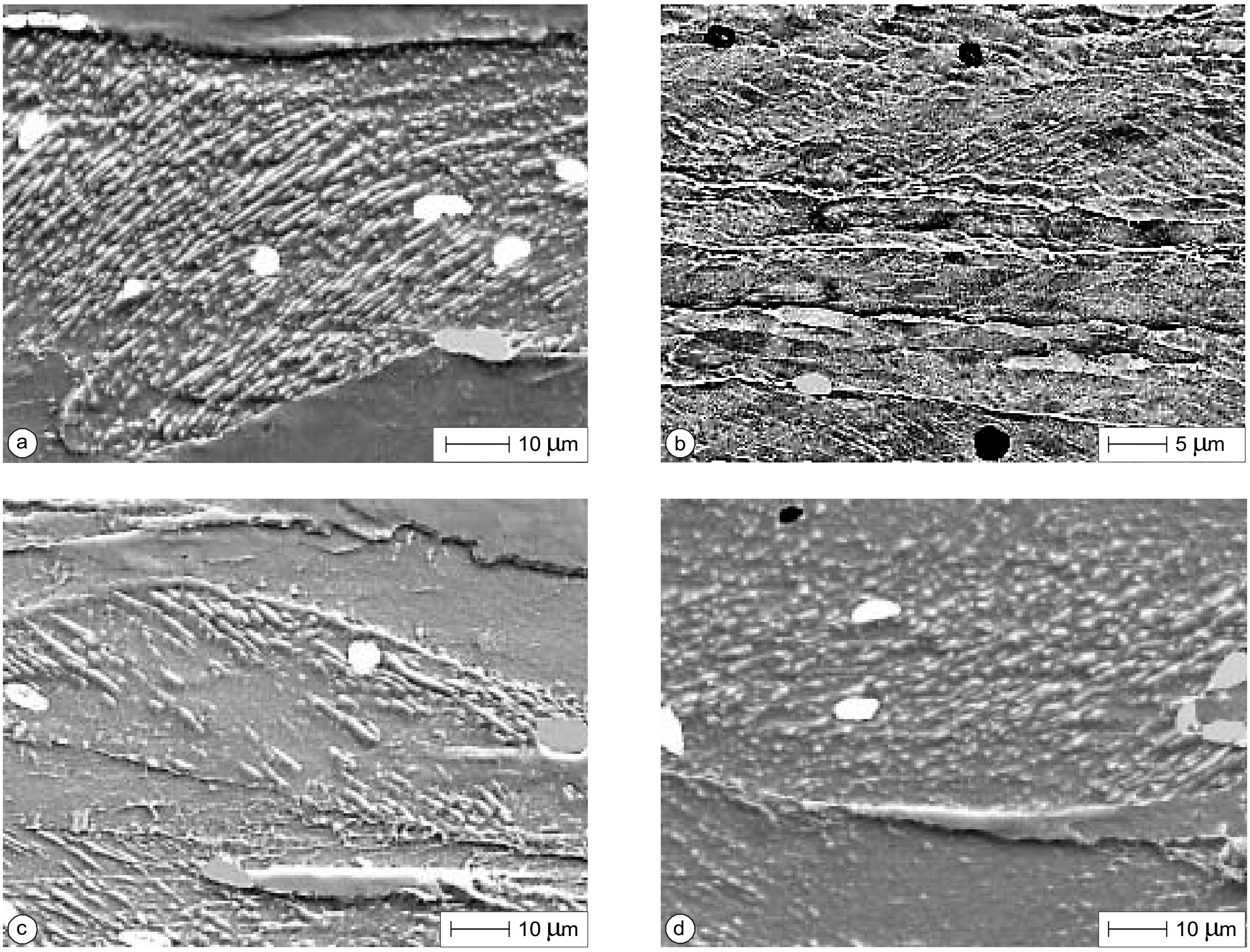

Figure 2. Representative SEM micrographs of the locations of nuclei in the 15 % recrystallised materials. (a) IF, (b) ELC, (c) LC(Cr), and (d) LC(Cr,P) (γ fibre is white, α fibre is grey and Goss is black)

Slika 2. Reprezentativni SEM mikrografovi lokacija nukleusa u 15-postotno rekristaliziranim materijalima. (a) IF, (b) ELC, (c) LC(Cr) i (d) (LC(Cr,P) (γ vlakno je bijelo, α je sivo, a Goss je crn)

Analysis of the evolution of average grain size of the recrystallisation nuclei has revealed the dependence of the growth rate on both the location of the nucleus and the stage of recrystallisation. The growth rate of grains nucleated at grain boundaries was higher than of the ones at shear bands at 5 % recrystallisation (Table 2.). The average size of the grain boundary nuclei at this stage was 3-4 times that of the shear band nuclei. With further progress of recrystallisation, the growth rates of the grain boundary nuclei decreased while the growth rates of those at the shear bands increased (Table 2.). The nucleation of new grains within the pearlite colonies was observed at a later stage ($\geq$15 % volume fraction recrystallised) than at the grain boundaries or shear bands. These nuclei were characterized by moderate grain sizes (Table 2.). The pearlite appeared to decompose during the early stages of recrystallisation, leading to the formation of spherical carbides; these carbides then became the nucleation sites for the recrystallised grains (Figure 3c).

The φ_2= 45° ODF sections for the IF, ELC, LC(Cr) and LC(Cr,P) steels for the 15 % and 100 % recrystallised conditions are given in Figure 4. Three fibres can be observed in all the steels: the α or RD fibre (<110> axis parallel to the rolling direction); the γ or ND fibre (<111> axis parallel to the normal direction); and the ε or TD fibre (<110> axis parallel to transverse direction). In the IF steel, the intensi-

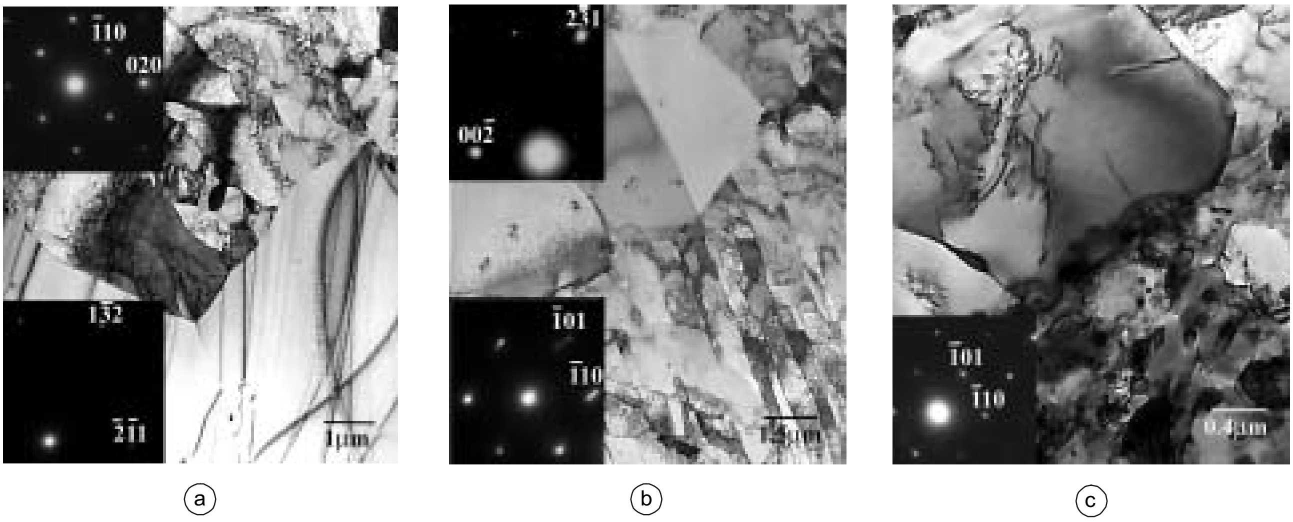

Figure 3. Examples of the locations of recrystallisation nuclei: (a) at grain boundaries (zone axis of the microbands is [001] and zone axis of the recrystallised grain is [157]); (b) at shear bands (zone axis of the shear bands is [111] and zone axis of the recrystallised grain is [320]); and (c) in a pearlite colony (zone axis of the recrystallised grain is [111]). Arrow indicates the grain boundary

Slika 3. Primjeri lokacija rekristalizacije nukleusa: (a) na granicama zrna (zona osi mikro trake je [001] i zona osi rekristalizacije zrna je [157]; (b) na smičnim trakama (zona osi smičnih traka je [111], a zona osi na rekristaliziranom zrnu je [320]; i (c) u perlet nakupini (zona osi rekristaliziranog zrna je [111]). Strijela pokazuje na granicu zrna

ties of the ND fibre orientations ({111}<110> and {111}<112>) increased during the progress of recrystallisation (Figures 4a and 4e); i.e., the ND fibre appeared to consume the RD fibre as the volume fraction recrystallised increased. The IF steel also had the highest volume fraction of ND fibre and the lowest of RD fibre at all stages of recrystallisation compared to the other steels (Table 3.).

Table 3. **Evolution of texture components during recrystallisation**

Tablica 3. **Razvitak komponenata teksture tijekom rekristalizacije**

Steel	Stage of Recrystallisation							
	5 %		15 %		50 %		100 %	
	%ND	%RD	%ND	%RD	%ND	%RD	%ND	%RD
IF	67	22	72	18	76	16	88	7
ELC	41	44	41	47	43	56	27	61
LC(Cr)	46	41	45	44	48	42	31	55
LC(Cr, P)	48	45	49	43	43	55	33	58
Balance: Goss and random components								

By contrast, the RD fibre was *strengthened* during recrystallisation in the ELC steel while formation of the ND fibre was suppressed. Among the four steels, it had the lowest number of γ-fibre grains at all stages of recrystallisation (Table 3.). At 15 % recrystallisation, the ELC steel already contained a strong RD fibre running from {001}<110> to {112}<110> (Figure 4b), which remained as the main fibre component after the completion of recrystallisation. The final texture was also characterized by the undesirable Goss component ({011}<100>), while the desirable ND fibre ({111}<110-112>) decreased in intensity (Figure 4f).

The addition of chromium and chromium-plus-phosphorus only increased the fraction of γ-fibre grains during the early stages of recrystallisation (up to 50 % volume fraction, see Table 3.). It can clearly be seen that the ND fibre intensity in the 15 % recrystallised samples has increased (Figures 4c and 4d). However, an increase in the annealing time led to the marked *deterioration* of the ND fibre and to increases in the intensities of both the RD and Goss components (Figures 4g and 4h). The ND component in the recrystallised LC(Cr,P) samples was slightly more intense than in the LC(Cr) steel (Figures 4d, h and Table 3.).

EBSD analysis revealed the relationship between the locations of the nuclei (in the microstructure) and their orientations. In all the steels, the shear band nuclei were preferentially of γ-fibre orientation, although some Goss component grains were also observed in the ELC and LC(Cr,P) steels (Figures 2b and 2d). The grain boundary nuclei in all the steels were preferentially of RD fibre orientation, although some belonged to the ND fibre (Figure 2.). Finally, the nuclei within the pearlite colonies had random and Goss orientations.

DISCUSSION

The recrystallisation kinetics of the present four steels have been measured in an attempt to clarify the mechanisms of formation of the final recrystallisation texture. The ELC steel displayed the highest rate of recrystallisation, which can be ascribed to the grain thickness being the finest (~ 10 μm) in the deformed structure of this material [9]. A grain thickness decrease increases the stored energy as well as the number of potential nucleation sites, thus accelerating the recrystallisation process in this way [10]. In the Cr- and Cr/P-alloyed low carbon

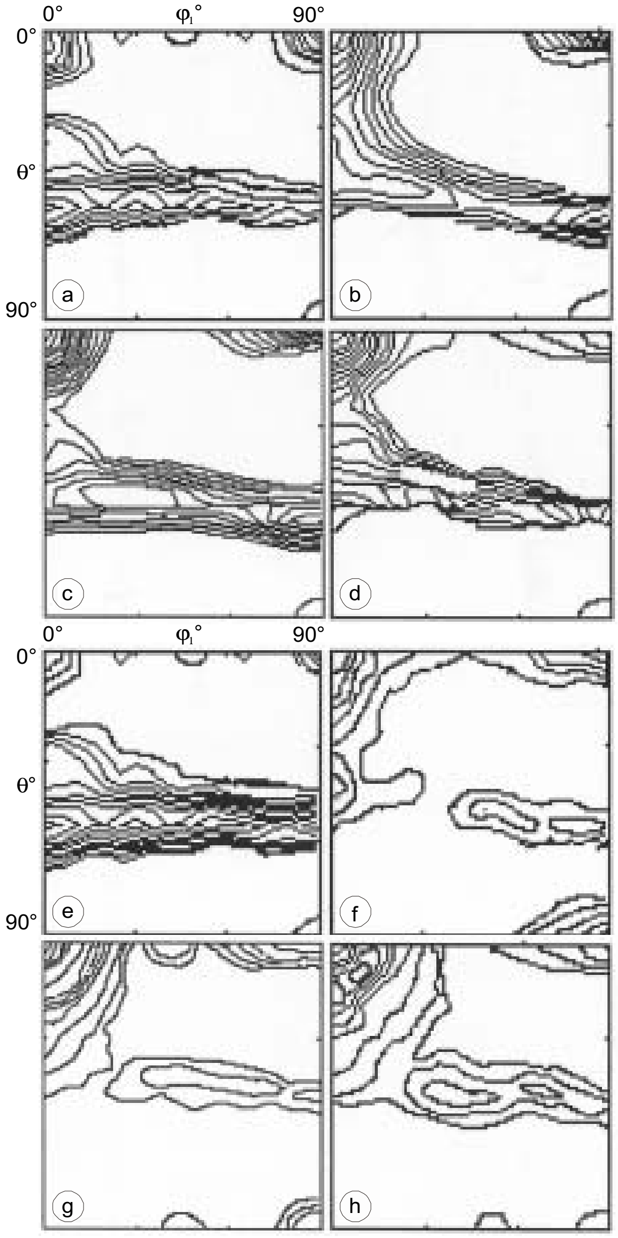

Figure 4. **φ_2 = 45° ODF's of the recrystallisation textures of the four steels: (a, e) IF, (b, f) ELC, (c, g) LC(Cr), and (d, h) LC(Cr, P). Recrystallised fractions of 0,15 (a, b, c and d) and 1 (e, f, g and h) are shown**

Slika 4. **φ_2 = 45° ODF rekristalizacijske teksture četiri čelika: (a, e) IF, (b, f) ELC, (c, g) LC(Cr) i (d, h) LC(Cr, P). Pokazane su rekristalizacijske frakcije 0,15 (a, b, c i d) i 1 (e, f, g i h)**

steels, the kinetics of recrystalli-sation were hindered by the following factors: i) the greater ferrite grain size after warm rolling (~ 20 μm) compared to the ELC steel [9] (which reduces the number of grain boundary nucleation sites); and ii) the chromium carbides present in the deformed microstructure, which decrease the mobility of the grain boundaries during recrystallisation [10]. The IF steel displayed the slowest rate of recrystallisation. This is associated with the coarsest grain size (at least three times greater than in the other steels) [9], as well as the presence of Ti in solution.

Differences were detected in the development of the recrystallisation textures in the four steels. The results demonstrated the importance of the development of the ND fibre during *both* stages of recrystallisation: i.e. during nucleation as well as growth. The IF steel exhibited the highest number of shear band nuclei. The majority of these nuclei had γ-fibre orientations. Analysis of the bulk texture confirmed that the ND fibre was dominant from the earliest stages of recrystallisation until its completion. An increase in carbon content, as in the ELC steel, led to the occurrence of DSA during warm rolling, to high positive rate sensitivities, as well as to a reduced fraction of grains containing in-grain shear bands [2, 9]. This decreased the number of nuclei with ND fibre orientations in the early stages of recrystallisation. Increases in the annealing time further suppressed formation of the ND fibre and promoted formation of the RD fibre.

The formation of chromium carbides in the microstructures of the LC(Cr) and LC(Cr,P) steels removed carbon from solid solution and in this way slightly increased the fraction of nuclei formed at shear bands compared to the ELC steel. This effect led to a reinforcement of the ND component in the early stages of recrystallisation. However, the strength of the γ-fibre deteriorated with the progress of recrystallisation. The main reasons for this undesirable development appear to be: i) the restricting effect of the carbides on the migration of grain boundaries during growth, ii) the influence of the particles on nucleus rotation during annealing (and on nucleation more generally), and iii) the absorption of γ-fibre nuclei by other components during grain coalescence and growth.

CONCLUSIONS

The recrystallisation kinetics of four different steels were studied. The results show that:

1. The IF steel displayed the slowest recrystallisation kinetics, the highest number of nuclei formed at shear bands, and the gradual strengthening of the γ component during annealing;
2. The addition of chromium and chromium-plus-phosphorus to the ELC steel led to the formation of chromium carbides in the deformed microstructure that hindered the recrystallisation kinetics. The initially formed γ-fibre nuclei were absorbed by the RD fibre grains during the later stages of recrystallisation. The recry-

stallisation texture in these steels was an intermediate one between the textures of the IF and ELC steels.

3. The addition of Cr is a promising route for the enhancement of the ND fibre during the nucleation stage of recrystallisation. However, to obtain the desired texture in a fully recrystallised state, further study of the texture behaviour during grain coalescence and growth is required.
4. The presence of carbon is detrimental to the production of desirable textures in several ways: i) it increases the volume fraction of pearlite (and thus the number of "random" nuclei); it increases the C solute content (and therefore reduces the extent of shear band nucleation); and iii) it increases the amount of carbide present and in this way hinders growth of the {111} component.

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