Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 16
ISSUE 1-2
May-June
2006

Refinement of steel microstructure by free-forging

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Received 15.11.2005; accepted in revised form 15.04.2006

Materials

ABSTRACT

Purpose: Refinement of austenitic steel and low alloyed C-Mn-Si-Nb steel microstructure by free-forging.
Design/methodology/approach: In this study severe hot deformation was applied on low alloyed 0.2C-1.5Mn-2Si-0.05Nb steel and austenitic 0.07C-18Cr-2Mn.-10Ni steel. Steels were processed in a full-hydraulic press. Different temperatures of preheating, dwells and strain levels were applied. Microstructure was characterised using optical metallography, scanning and transmission electron microscopy.
Findings: After optimization of processing fine grained structures with convenient phase composition were obtained. Multiple free-forging followed by recrystallization annealing was successfully used for refinement of austenitic Cr-Ni steel and improvement of mechanical properties. While free-forging of low alloyed C-Mn-Si-Nb steel still has not satisfied expected increase of strength and ductility.
Research limitations/implications: Free-forging usually results in formation of very heterogeneous structures. In submitted study relatively fine grained and uniform structures were obtained. Grain size below a few micrometers can not be probably achieved using above mentioned technology.
Originality/value: Obtained results can be used for development of forging technology in service conditions.
Keywords: Fine-grained structure; Austenitic steel; Microalloyed steel; Free forging; Microscopy

1. Introduction

Refinement of microstructures in steel has received considerable attention in recent times because it results in remarkable improvement of mechanical properties. Small grain size corresponds to a large area of grain boundaries and increases yield stress, ultimate tensile strength, notch toughness and ductility.

One of the effective methods of microstructure refinement is severe plastic deformation, which can be applied using different forming technologies. Controlled rolling [1] is still the most successful method that enables to obtain uniform fine-grained structure nearly in the whole volume of plate. Other advanced metal-forming technologies, such as equal channel angular pressing [2] or extrusion with torsion and bending [3,4], are being developed.

Free-forging is commonly used to increase strength values; however it is rarely used in studies of ultrafine-grained materials. The reason consists in difficulties to ensure the same thermal and strain conditions in the whole volume of processed part if compression deformation is applied. Consequently microstructures and also macroscopic properties in different parts of forged component usually differ and cracking can occur under loading.

Exceptional combination of high strength and ductility can be obtained in the case of multistructural low alloyed steels, if the effect of transformation induced plasticity is employed. Utilization of TRIP effect is conditioned by convenient phase composition, uniform distribution of individual structural components and small grain size. Usually mixed ferrite-bainite microstructure is required with high content of retained austenite. Austenitic islands have to be dispersed at the grain boundaries of fine-grained proeutectoid ferrite and in bainitic ferrite. Different TRIP steels were developed on the base of low alloyed manganese steels with a high content of silicon and microadditions of niobium, aluminium, cuprum or phosphorus [5]. For their production special thermo-mechanical processing has to be used [6].

Table 1.
Forging procedures applied on austenitic steel

Specimen	Temperature of preheating [°C]	Thickness of specimen after individual reductions [mm] (rotation of specimen) 1 (90°)	2 (45°)	3 (90°)	4 (45°)	5 (90°)	6 (45°)	Dwells between blows [s]
A1	1050	12	17	12	17	12	16	3
A2	1150	10	15	10	15	10	15	3
A3	1150	10	15	10	15	10	15	3
A4	1050	10	15	10	15	10	15	6
A5	1100	10	15	10	15	10	15	6
A6	1150	10	15	10	15	10	15	6

In this paper preheating at different temperatures and multiple free-forging operations were applied to two materials: austenitic Cr-Ni stainless steel and multistructural low alloy Mn-Si-Nb steel with the aim to obtain as fine-grained as possible and uniform microstructure. Microstructural investigation and mechanical testing were carried out.

2. Austenitic stainless steel

2.1. Experimental procedure

The chemical composition of steel used was 0.07 C, 18.0 Cr, 2.0 Mn, 10 Ni, 0.045 P, 0.030 S, 1.0 Si (wt%). Continuously cast bars were forged and annealed at 1100°C/ 1.5 hours. Cylindrical specimens with a diameter of 20 mm and a length of 55 mm were machined and preheated at different temperatures for 40 minutes in furnace. Drawing out was applied using full-hydraulic press CKW 600 (Fig. 1). Different dwells between individual blows and strain levels were carried out. Specimens were rotated after each of blows at 90° and at 45°. Immediately after forging water cooling followed (Table1).

Each of processed specimens was cut along its transversal axis and metallographic sample was produced. Microstructure was revealed by electrolytic polishing and etching in 60 % nitric acid and observed in light (LM) and scanning electron microscope (SEM – JEOL JXM 840).

Carbon extraction replicas and thin foils were prepared for transmission electron microscopy (TEM). Foils were thinned to electron transparency in 6% solution of $HClO_4$ in CH_3OH at the temperature of – 40°C. TEM was performed at the accelerating voltage of 120 kV using microscope JEOL JEM 1200 EX.

Two specimens with the most promising microstructure were cut into slices with a thickness of 3 mm and recrystallization annealing was carried out at temperatures of 750°C, 850°C or 950°C for 0.5 hour or 2 hours.

After austenitization at 1050°C, 1100°C or 1150°C six blows with different dwells were applied. Forging procedures of individual specimens are described in Table 1. Processing of specimens A3 and A4 was identical for the purpose of verification of its reproducibility.

Microstructure of all specimens after multiple hot compression deformation was very heterogeneous. Different stages of recrystallization were observed depending on local strain level and temperature during forging [7]. In central parts of specimens a fine-grained fully recrystallized structure was present, while in remaining parts partly recrystallized structure was observed.

Specimens A4 and A6 had the highest volume fraction of recrystallized microstructure. Longer dwells between individual blows enabled postdynamic processes and consequently microstructure in large regions was refined.

Thin foils and carbon replicas were prepared from specimen A6. In the fine-grained central part austenite grains at different stages of plastic strain were present as a result of dynamic recrystallization and recovery during the last forging procedure.

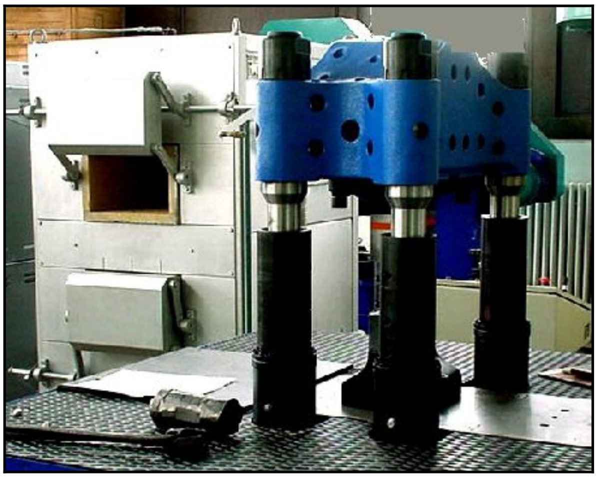


Fig. 1. Full-hydraulic press CKW 500

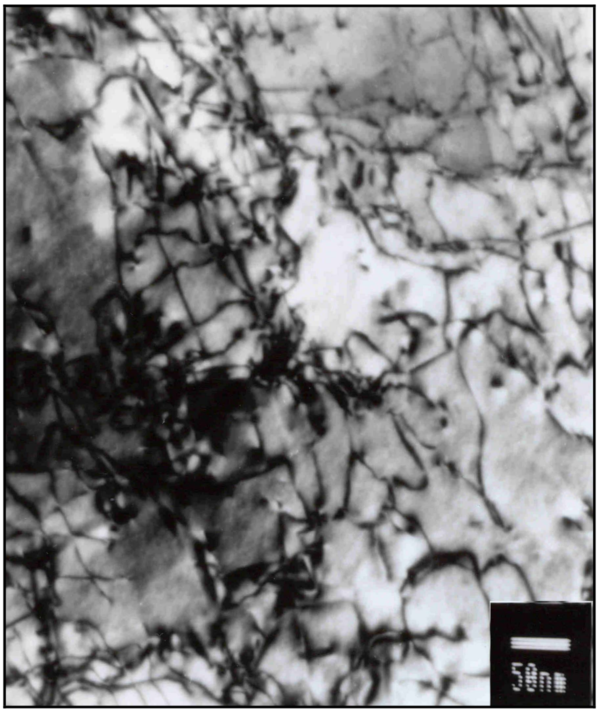


Fig. 2. Central part of specimen A6. TEM micrograph of thin foil. Strained austenite grain with a high dislocation density.

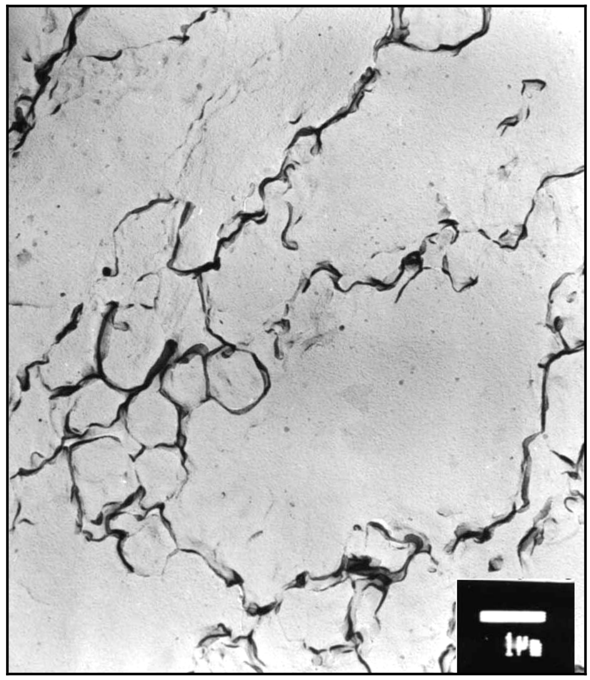


Fig. 4. Partly recrystallized structure of specimen A6.TEM micrograph of extraction carbon replica. Large strained and small recrystallized grains.

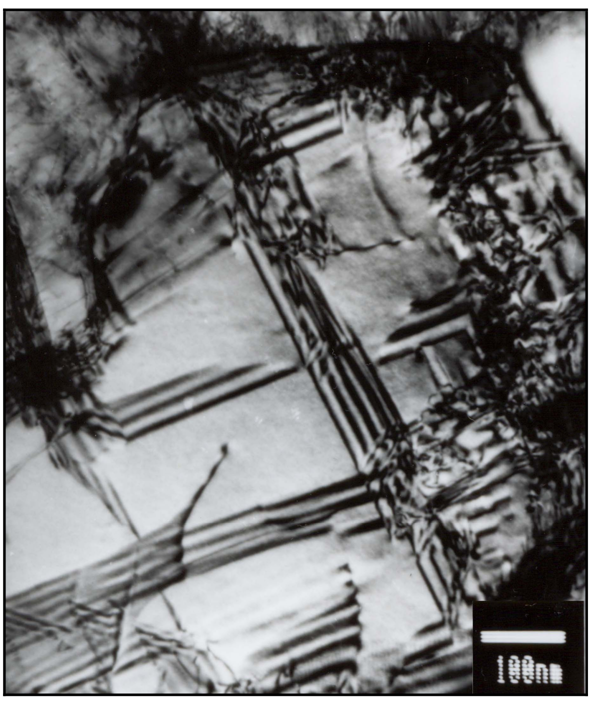


Fig. 3. Central part of specimen A6. TEM micrograph of thin foil. Strained austenite grain with deformation twins.

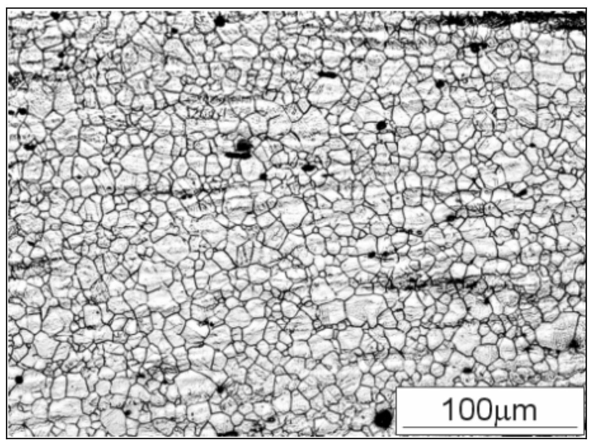


Fig. 5. Specimen A6 after annealing 850°C/0.5h, LM micrograph of metallographic sample after electrolytical etching.

Both fundamental deformation modes slip and twinning occured in the steel used in this study. Most of the grains were deformed by slip [8]. A high and also very low dislocation density was observed (Fig. 2). Some of the grains were strained predominant by twinning (Fig. 3). It depends on crystallographic orientation of individual austenitic grain and the local strain conditions, which of deformation modes occurs [9].

Using selected electron diffraction small δ-ferrite grain was identified. Any precipitates were found in foils and on extraction replicas. In partly recrystallized regions were observed small austenite grains distributed on grain boundaries of larger austenite grains (Fig. 4).

Specimens A4 and A6 underwent subsequent recrystallization annealing. Fully recrystallized microstructure in the whole volume of specimens was obtained after annealing at 850 °C [10]. Finer-grained microstructure was observed if 0.5 hour dwell at annealing temperature was used (Fig. 5).

Recrystallized grains with a low density of crystal defects were revealed in scanning electron microscope using electron channeling contrast (Fig. 6). These SEM micrographs were used for grain size measurement in image analysis program Lucia; results are shown in Table 2.

When annealing at 750°C was applied recrystallization was not completed, after annealing at 950°C coarse-grained structures were observed.

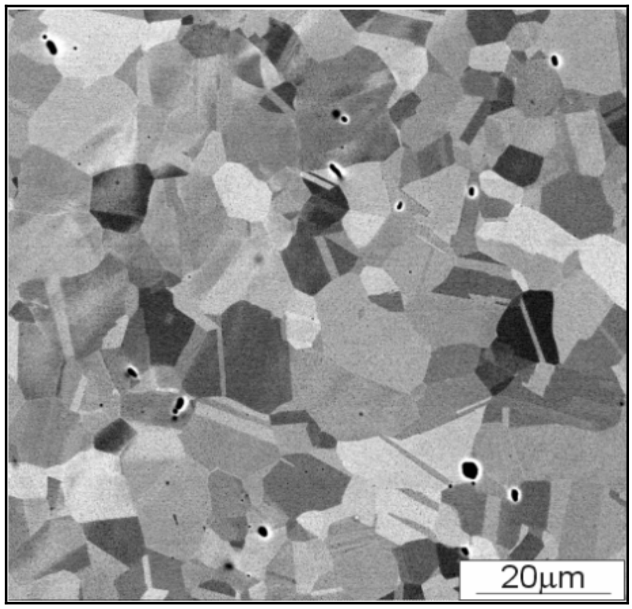


Fig. 6. Specimen A6 after annealing at 850°C / 0.5h. SEM micrograph – backscattered electron image of metallographic sample after electrolytic polishing.

Table 2.
Grain size after recrystallization annealing

Specimen	Annealing temperature [°C]	Dwell [h]	Grain size [μm]
A4	850	0.5	8,1
A4	850	2.0	15,9
A6	850	0.5	7.8
A6	850	2.0	9.4

3. Low alloy steel

3.1. Experimental procedure

The second material investigated in this study was a trial heat of low alloy steel having the chemical composition 0.19 C, 1.39 Mn, 1.92 Si, 0.012 P, 0.006 S, 0.04 Cr, 0.04 Cu, 0.026 Al, 0.09 Nb and 0.015 N (wt%). Cast bars were preheated in laboratory furnace to the temperature of 1100°C and than forged using V-die. Cylindrical specimens were machined out with a diameter of 18 mm and a length of 60 mm.

Microadditions of niobium in steels result in formation of carbonitride precipitates. They can inhibit grain growth during austenitization and also can contribute to precipitation strengthening. Niobium atoms in solid solution retard ferritic and pearlitic transformation during austenite decomposition, increase hardenability and stability of retained austenite. For these reasons different austenitization temperatures were used before subsequent processing in hydraulic press.

The specimens were preheated in the furnace at austenitization temperatures 980°C or 1230°C. Multiple free-forging consisted of different dwells, strain levels and numbers of blows. Between individual blows specimens were rotated at 90°. After forging specimens were cooled in water or in the air. Sequences of individual forging procedures were developed on the base of previous simulations of thermo-mechanical processing using simulator SMITWELD TTU 2002, which enables to control temperature and strain conditions [11,12].

After forging microstructural analyses were carried out. All specimens were cut along their longitudinal and transversal axis and metallographic samples were prepared. Microstructure was revealed by conventional etching in 3% nital and also by colour etching in 10% aqueous solution of $Na_2S_2O_5$. Volume fraction of ferrite was evaluated from LM micrographs using image analysis program Lucia. Volume fraction of retained austenite was established by X-ray diffraction phase analysis and also by magnetometric method. From the etched metallographic samples carbon extraction replicas were prepared and TEM was performed. Mechanical properties were tested using Charpy and tensile tests.

3.2. Results

The first set of specimens was austenitized at 980°C for 1 hour. Individual forging procedures were optimized step by step using hydraulic press. At the beginning only one 50% compression deformations was carried out in austenitic region. Then different dwells were hold and water quenching followed. In martensitic microstructure was measured the grain size of prior austenite. The mean grain size in central parts of different specimens ranged from 8.5 μm to 9.7 μm.

Then attention was paid to forming in dual phase region. After optimized first forging procedure one or two blows with different dwells were applied and again water quenching followed. Microstructure consisted of ferrite and martensite. On the base of these experiments two promising forging methods were selected, which resulted in convenient content of ferrite; volume fraction of ferrite in central parts of specimens corresponded to 45% and 60%. Forging was completed by dwell in bainite region.

Schedule of optimized free-forging with preheating at 890°C is described hereafter.

<u>Specimen T1:</u>
austenitization 890°C/ 1h in furnace,
5 s dwell in the air,
50% deformation, 16 s dwell in the air,
50% deformation, 8 s dwell in the air
30% deformation, 10 s dwell in the air,
water cooling to 410°C, 300s dwell in the air,
cooling in the air.

<u>Specimen T2:</u>
austenitization 890°C/ 1h in furnace,
5 s dwell in the air,
50% deformation, 24 s dwell in the air,
50% deformation, 17 s dwell in the air,
water cooling to 410°C, 300 s dwell,
cooling in the air.

a)

b)

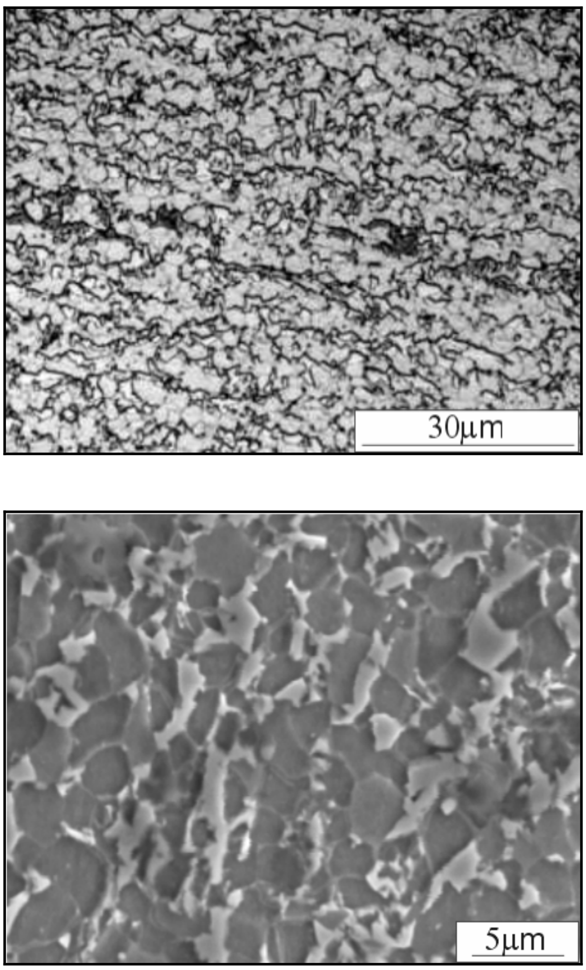


Fig. 7. Central part of specimen T1, metallographic sample, colour etching: (a) LM micrograph, (b) SEM micrograph – secondary electron image.

Table 3.
Volume fraction of retained austenite

Specimen	V_F [%]	V_{Ax} [%]	V_{Am} [%]	UTS [MPa]	E [%]
T1	45	38	16	786	21
T2	60	21	15	839	21

V_F …volume fraction of ferrite (image analysis), V_{Ax}…volume fraction of retained austenite (X-ray diffraction), V_{Am}… volume fraction of retained austenite (magnetometry), UTS…ultimate tensile strength, E…elongation.

a)

b)

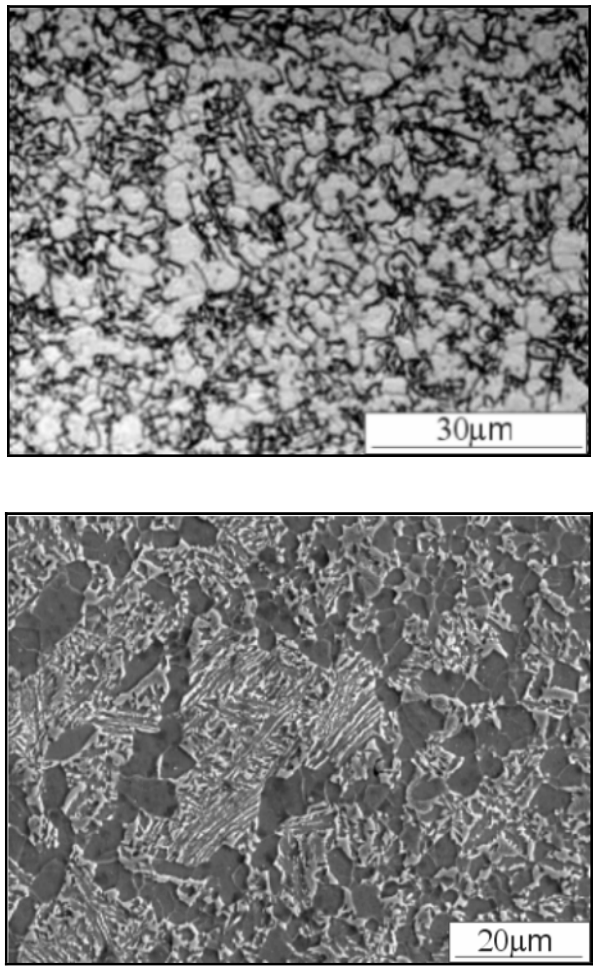


Fig. 8. Rim part of specimen T1, metallographic sample, colour etching: (a) LM micrograph, (b) SEM micrograph – secondary electron image.

Microstructure of specimens T1 and T2 consisted of fine-grained proeutectoid ferrite, islands of retained austenite on ferrite grain boundaries and granular bainite, which was formed from bainitic ferrite and retained austenite between individual laths. In fine-grained central parts proeutectoid ferrite and retained austenite predominately occurred (Fig. 7), in rim regions were also present bainitic blocks (Fig. 8). Results of quantitative microstructural evaluation are summarized in Table 3.

Forging methods of specimens T1 and T2 were applied on additional specimens, which were used for mechanical testing (Table 3). Higher ultimate tensile strength was achieved for specimen T2, elongation was the same for both specimens. Microstructural analyses of tensile test specimens confirmed TRIP effect. Retained austenite transformed to martensite.

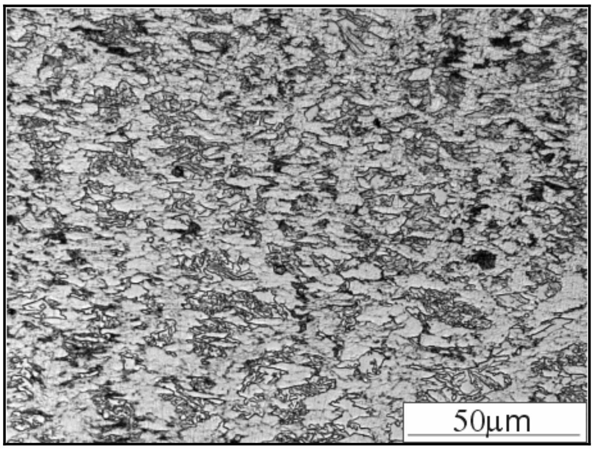


Fig. 9. Central part of specimen T3, LM micrograph of metallographic sample etched in nital.

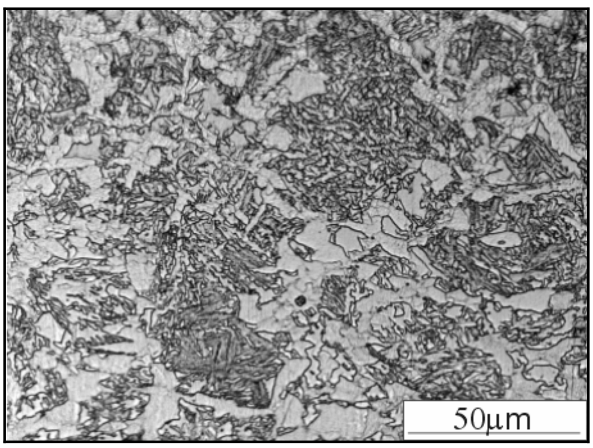
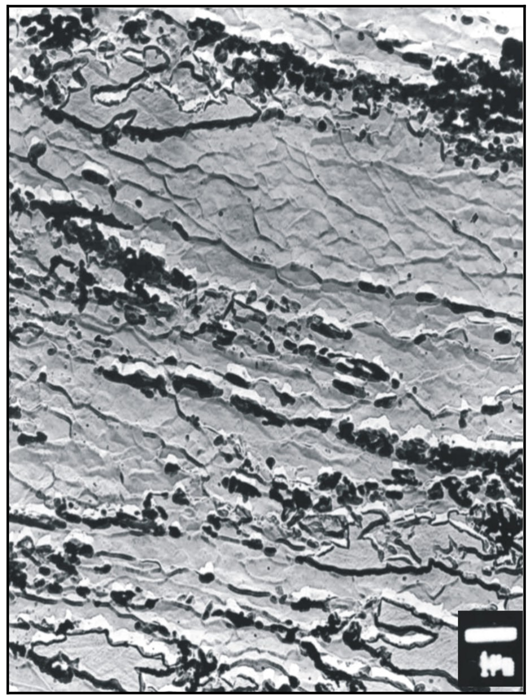


Fig. 10. Rim of specimen T3, LM micrograph of metallographic sample etched in nital.

The second set of specimens was autenitized at a temperature of 1230°C, which enables dissolution of niobium carbonitrides. Forging procedures were optimized in successive steps using thermo-mechanical processing (TMP) in SMITWELD simulator [11,12]. The schedule of real forging is based on optimized TMP-method and was calculated using numerical simulations. It consisted of furnace austenitization at 1230°C for 5 minutes and four 50% compression deformations. Two deformations were applied at 1100°C, third deformation at 750°C and the fourth one at 680°C. The specimen (labelled T3) was rotated at 90°during individual blows. Pressure air cooling to 410°C was carried out immediately after the last deformation and 300 s dwell in furnace at 410°C followed. Processing was finished by air cooling.

a)



b)

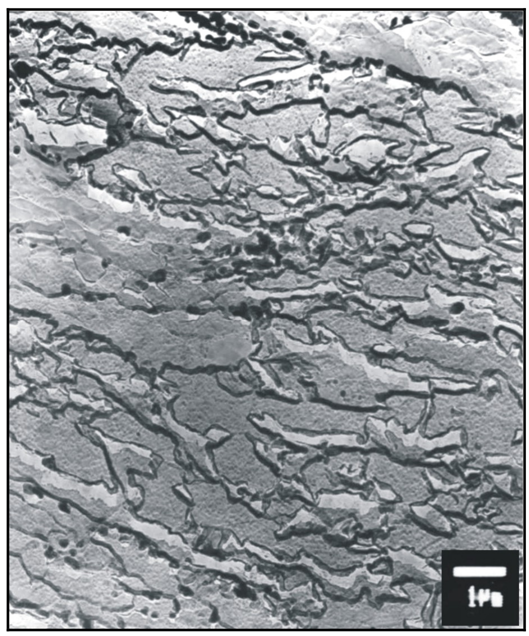


Fig. 11. Specimen T3, TEM micrographs of carbon replicas: (a) central part, (b) rim part.

Microstructures in different regions of specimen T3 were studied using metallographic samples and carbon replicas. Fine-grained structure in central part consisted of proeutectoid ferrite and bainite with sporadic presence of small pearlitic areas (Fig. 9). Bainitic blocks were growing toward the rim of specimen (Fig. 10). In narrow band along longitudinal axis of specimen were observed many carbide particles. They formed rows in localities, where slip bands were concentrated during the last deformation (Fig. 11a). In some rows with a high density of carbides were also observed nodules of lamellar pearlite. Remaining parts of specimen contained ferrite and retained austenite (Fig. 11b). Carbide particles occurred only exceptionally outside the central band.

Mechanical properties of specimen T3 were established with the use of tensile test and Charpy test. Ultimative tensile strength of 1057 MPa and 13% elongation were achieved. Impact toughness KVC = 40 J/cm^2.

4. Discussion

Microstructure of bars made from Cr-Ni austenitic steel was very heterogeneous after free-forging; plastic strain was concentrated in central parts and in slip bands along diagonals of specimens. Rotation of specimen between individual blows contributes to iniciation of slip in new crystallographic systems, nevertheless in rim parts of specimens strain level remains low. This non-uniform strain level distribution is caused by a high friction between specimens and dies. Friction forces could be reduced using lubrications or shaping of contact planes of dies and specimen. However both methods can not be applied for multiple free-forging.

Microstructural study of processed specimens demonstrated that recrystallization was completed only in central fine-grained parts of specimens processed in hydraulic press. In rim parts partly recrystallized structures were observed. Different dislocation density and also number of deformation twins were observed in individual austenitic grains.

When recrystallization annealing at 850°C for 0.5 hours was applied, uniform microstructure was obtained in the whole volume of specimen. Mean grain size of 7.8 μm was achieved. Such a microstructure guarantees considerable improvement of mechanical properties similar to results published in [13].

Multiple free-forging applied on low alloy steel resulted in formation of desirable phase composition. Microstructure consisted of proeutectoid ferrite, bainite and retained austenite.

Volume fraction of individual structural components fulfilled requirements on microstructure of TRIP steels: ferritic-bainitic microstructure with (40 ÷ 60) % of proeutectoid ferrite and (5 ÷ 15) % of retained austenite. Microstructure of the steel used in this study contained 60% of proeutectoid ferrite and a high amount of retained austenite. Values of austenite fraction determined by X-ray diffraction and by magnetometric measurement differ considerably. Higher values were obtained by X-ray diffraction measurement, which occurred only in central parts of specimens, were majority of dual ferrite-austenite microstructure was observed. While magnetometric measurement represented the whole volume of specimens. Therefore the last mentioned measurement method provides more representative values of austenite fraction in specimens used in this study.

Concerning the size and distribution of individual structural components are obtained results not satisfying. Sufficiently fine-grained microstructure occurred only in central parts of specimens. Grain size of ferrite only slightly increased proportionally to distance from the longitudinal axis of specimen, however the size of bainitic areas increased much rapidly. Large bainitic blocks probably caused that expected improvement of mechanical properties was not achieved.

When the steel with similar chemical composition as steel used in this study underwent forming by controlled rolling ultimate tensile strength of 890 MPa and elongation of 26 % were achieved, while for Mn-Si steel without niobium microaddition 970 MPa and 43% were obtained [14]. In this study the best values corresponded to 839 MPa and 21%.

If we compare results of forging with preheating at 980°C and 1230°C, it is evident that lower austenitization temperature is more convenient for microstructure refinement. Primary niobium carbonitrides restrict grain growth during hot deformation in austenitic region and also during phase transformation to ferrite and bainite.

Positive influence of niobium atoms dissolved in solid solution was not confirmed. After optimized processing with austenitization temperature of 1230°C ultimate tensile strength UTS = 1057 MPa was achieved, however ductility was very low (E = 13%). Stabilizing effect of niobium on retained austenite, which would result in increase of ductility, was not proved. Obtained high strength values can be caused not only by deformation induced transformation of austenite to martensite but also by fine niobium carbonitrides, which could precipitate in ferrite after austenite decomposition. As yet these small particles have not been observed on carbon replicas.

5. Conclusions

Multiple free-forging was successfully used for refinement of austenitic Cr-Ni steel and improvement of mechanical properties. While its utilization for low alloy Mn-Si-Nb steel did not satisfy expected increase of strength and ductility.

Aknowledgements

This work was supported by a Grant project of Faculty of Mechanical Engineering of West Bohemia University in Pilsen.

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