Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 18
ISSUE 1-2
September–October
2006

Strength enhancement possibilities of low carbon steels

M. Greger [a,*], R. Kocich [a], L. Kander [b], G. Dorociáková [a]
[a] Faculty of Metallurgy and Materials Engineering,
VSB-Technical University of Ostrava, Czech Republic
[b] VÍTKOVICE - Research and Development, Ltd., Ostrava Vítkovice, Czech Republic
* Corresponding author: E-mail address: miroslav.greger@vsb.cz

Received 15.03.2006; accepted in revised form 30.04.2006

Manufacturing and processing

ABSTRACT

Purpose: The paper analyses methods of grain refinement and demonstrates development of structure and properties of metallic materials after severe plastic deformations (SPD).
Design/methodology/approach: Technology ARB was experimentally verified. The material was rolled in 11 passes. Rolling proceeded at temperature 650˚C, with heating in furnace with inert atmosphere (Ar).
Findings: True strain has achieved the value 9. Basic relations between magnitude of deformation, grain refinement and resulting mechanical properties were described. Bonding of degree was greater than 90%.
Practical implications: ARB method is one of instrument for acquirement materials with ultrafine grain structure. Is it very simply apparatus, which can be used in practical technology (classical rolling mill).
Originality/value: Development of structure was verified on low carbon steel. Obtained grain size was around 0.3 μm. Properties obtained by tensile test did not achieve the expected value. Grain size was homogenous in whole volume.
Keywords: Plastic forming; Ultra-fine grain structure; Mechanical properties

1. Introduction

Microstructural conditions for enhancement of strength properties of low-carbon steels and alloys consist of fine grain and its stability [1-5]. At present there are known several methods for grain refinement and for limitation of its growth. Phase transformation, re-crystallisation, severe plastic deformations (SPD), deformation of alloys with duplex structure and distribution of phases in duplex alloys can lead to grain refinement. Distribution of alloying elements between phases in duplex structures dispersively precipitated particles can be used for limitation of grain growth [6, 7]. Selection of methods of grain refinement and deceleration of its growth is given in individual cases by state and properties of the alloy. We will demonstrate hereinafter use of some grain refinement methods and deceleration of its growth for high-strength aluminium alloys.

Fine grain is an indispensable condition of enhancement of strength properties (Fig. 1) and for formation of superplasticity effect [8].

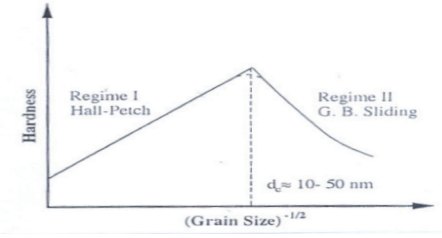


Fig. 1. Dependence of mechanical properties on grain size

Connection between superplasticity and fine grain follows from the substance of dependence of stress on strain rate. It is presumed that slipping along grain boundaries and diffusion creep brings a substantial contribution into overall deformation at low strain rates and low values of stress. Slipping along grain boundaries is the principal mechanism of superplastic deformation [9-11], that's why it is necessary to obtain small grain size and to slow down it's coarsening during deformation at high temperatures.

Deformation realised by movement of dislocation requires substantially higher stress, than deformation realised by mechanism of slipping along grain boundaries.

2. Grain refinement methods

Several methods are used for grain refinement, such as phase transformation, re-crystallisation, forming of duplex alloys and distribution of phases in duplex alloys. In case of the first two methods – phase transformation and re-crystallisation the mechanism of these processes is based on formation of nuclei of new grains inside the grains of initial structure.

In the third method – deformation of duplex alloys, both phases disintegrate and coagulate. Process in both phase is accompanied by re-crystallisation, which brings another contribution into overall grain refinement.

In case of the fourth method the initial structure is not an equilibrium microstructure. It can be e.g. martensite, or over saturated solid solution. Distribution of metastable phase to two equilibrium phases leads to formation of ultra-fine grain duplex structure. Very often several of these methods are used simultaneously. Diagram in Fig. 2 show four grain refinement methods mentioned above.

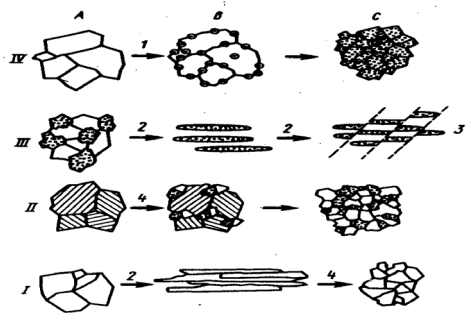


Fig. 2. Basic mechanisms of grain refinement I – re-crystallisation; II – distribution of phases in alloys with duplex structure; III – non-homogenous deformation of alloys with duplex structure; IV – phase transformation; A, B, C- initial, intermediary and final microstructure 1 - Heating cooling; 2 – deformation; 3 – shear planes; 4 – annealing

2.1. Grain refinement by phase transformation

Transformation of ferrite to austenite is used for grain refinement in steels**.** It was demonstrated that cycling of temperature near transformation temperature leads to formation of very fine grain. The smaller is the grain during next cycles, the bigger number of nuclei is formed for subsequent transformation. At ultra small dimension of grains next cycling will not influence grain size. Method of controlled forming aimed at obtaining of fine grain was developed for high-strength low-alloyed steels.

Mechanism of grain refinement at controlled forming consists of transformation of heat strengthened non re-crystallised austenite into ferrite [12]. Carbo-nitrides of niobium and vanadium determining re-crystallisation of austenite are precipitated in the course of deformation. Severely deformed austenite ensures high density of places of nuclei of ferrite grains and these grains are after transformation very fine.

2.2. Grain refinement of alloys with duplex structure by deformation

Selected superplastic alloys have duplex structure, i.e. that they have two formable phases. These are e.g. titanium based alloys, hyper-eutectoid steels, etc. In case of alloys with duplex structure it is possible to use two methods of grain refinement:

- deformation of alloy with uniform duplex microstructure;
- re-crystallisation of alloys with non-uniform structure.

Our paper analyses the possibility of grain refinement by deformation, namely in alloys with equilibrium duplex structure. Deformation is followed by annealing in case that dynamic re-crystallisation occurs during deformation. Uniform deformation of alloys with duplex structure supports forming of longitudinal particles of phases, preferably re-crystallised, in such manner, that each initial grain contains several new grains, but phases at the same time cannot be shifted. Phases do not intermix, they have different chemical composition, which means that: α- phase at re-crystallisation forms an α- phase, β- phase from a β- phase.

The key moment at grain refinement in alloys, which already have duplex structure, is non-uniformity of deformation. Pictures of deformed duplex microstructures show distinctly non-homogenous course of deformation. Areas with longitudinal phase disintegrate in the course of deformation and deformation is profoundly non-homogenous. By annealing it is possible to influence re-crystallisation of individual phases, running diffusion processes create conditions for spheroidisation and formation of the required equi-axed ultra-fine grain structure. At small deformations the initial grains are not sufficiently strengthened and equi-axed duplex microstructure will not be formed by re-crystallisation.

2.3. Grain refinement by decomposition of metastable phase (alloys with duplex structure)

Annealing of alloys with metastable phase causes its decomposition to two equilibrium phases ($\alpha + \beta$). Decomposition results in formation of fine-grained duplex microstructure. It is possible to obtain fine grain is the initial non-equilibrium phase contains high density of nuclei of α and β phases. Equilibrium mixture of two phases is obtained by diffusion processes. Material transfer by diffusion depends on chemical composition of equilibrium phases and on density of nuclei.

Initial non-equilibrium structure in steels is usually martensite or super-cooled solid solution. Presence of sub-structure in martensite increases density of nuclei of equilibrium phases. Deformation of super-cooled solid

solution before its decomposition increases the effect of grain refinement during decomposition.

Method of decomposition of metastable phase is used in titanium alloys [13]. Martensite, obtained by quenching from β zone, is initial structure. Annealing in (α + β) causes decomposition of martensite to equilibrium α and β phases. Degree of grain refinement depends on type of annealing, which controls speed of formation and growth of equilibrium phases.

At higher temperatures a transformation occurs in monotectoid alloys, which is running according to a classical scheme of eutectoid transformation, flat structures are formed with low superplasticity indicator. Eutectoid decomposition at room temperatures runs slowly and duplex microstructure is formed, which consist of two equilibrium phases. Spinoidal decomposition in binary alloys runs quickly and coherence of inter-phase boundaries is disturbed.

2.4. Grain refinement by re-crystallisation

Re-crystallisation is a universal method for grain refinement – unlike grain refinement methods described above, which are suitable only for alloys, in which transformation arise or for alloys with duplex microstructure. The results obtained during last years [14] declare influence of size of individual structural particles on the course of re-crystallisation. There are two types of effect of particles on re-crystallisation:

1. Fine particles (with diameter much smaller than 1 μm) inhibit nuclei and slow down growth of re-crystallised grains as a result of blocking of walls of dislocation cells and sub-grain boundaries.
2. Coarser particles (with diameter larger than 1 μm) create places of nuclei for re-crystallisation. During deformation deformed zones are formed around non-deformable particles and formation of nuclei of re-crystallised grains occurs inside these deformed zones. It is possible to use two types of re-crystallisation for grain refinement. Both are connected with use of particles for creation of fine grain.

Discontinuous re-crystallisation – nuclei are formed and re-crystallised grains grow. In this case the key factor is high density of nuclei for re-crystallised grains. It is obvious that high density of large particles will refine grains at running discontinuous re-crystallisation. This mechanism of grain refinement was verified on dispersively hardenable Al alloys. Mean diameter of grains was around 10 μm and alloys had high values of strength.

Continuous re-crystallisation – sub-grains grow, high-angle boundaries are formed and structure is transformed to re-crystallised structure. Nuclei of individual re-crystallised grains are not formed. Braking of discontinuous re-crystallisation by more rapid process is pre-requisite for running of continuous re-crystallisation. It is possible to expect continuous re-crystallisation in alloys with high density of fine particles, which prevent running of discontinuous re-crystallisation.

2.5. Grain refinement by severe plastic deformation

Several procedures are used (ECAP, S2C2, ARB), during which severe plastic deformation accumulates in bulk of metal. Magnitude of deformation at classical techniques of forming is limited by shrinking of material cross section with growing pass reduction. Technology ARB (accumulative roll bonding) is a SPD process, at which severe deformations are obtained by rolling [15]. The principle is shown in Fig. 3. It is possible to repeat the ARB almost without any limitations.

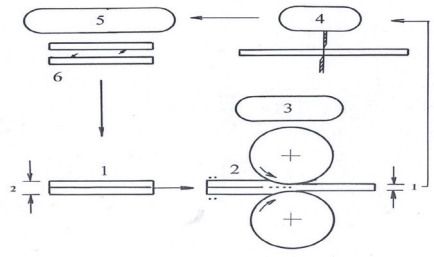


Fig. 3. Principle of ARB process 1 – initial sample, 2 and 3 – rolling, 4 – transverse cutting, 5 and 6 surface finish and stratification

3. Experimental verification

ARB process was verified in laboratory conditions on low carbon steel and on austenitic steel. Under given conditions of rolling certain problems occurred with pressure welding of individual layers in case of austenitic steel, that's why results of the experiment will not be presented in greater detail. Chemical composition of low carbon steel is given in the table 1.

Table 1.
Chemical composition of low carbon steel

Chemical composition [mass %]					
C	Mn	Si	P	S	Al
0.05	0.45	0.15	0.020	0.020	0.020

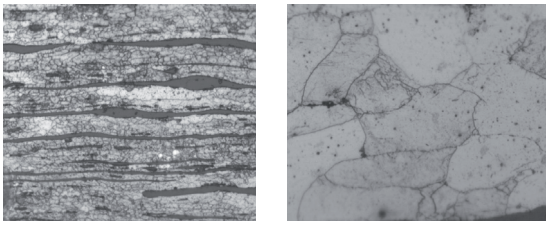
3.1. Results and their analysis

Altogether 11 cycles were made at temperature of 620 °C. Grain was gradually refined with increasing temperature. Development of structure is shown in Fig. 4. Structure is non-uniform and there are areas with grain size smaller than 0.5 μm. Boundaries between grains are distinct, but irregular. Similar structure can be found in severely deformed materials, obtained by different SPD processes.

Characteristic feature of materials processed by the ARB process is their orientated structure. Photos show typical microstructure of elongated very fine grains in steel after 11 cycles. Total number of individual sheets in the packet is 2 048.

It is obvious from photos of microstructures that UFG structure is not formed by sub-grains, but rather with grains with

high-angle boundaries. Maximum deformation obtained by the ARB process is e = 8,8. Grains are elongated in direction of rolling. It is possible to discover very similar microstructures also in other metals with KSC structure processed by the ARB process. These microstructures resemble to lamellar structures observed in severely deformed materials (Fig.5)



a) 500 x b) 500 x

Fig. 4. Grain size after deformation e = 2,4 *(a)* and for e = 8 *(b)*

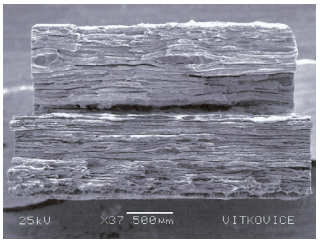


Fig. 5. Lamellar structure of low carbon steel after deformation e = 8.8

Table 2.
Properties of samples after deformation e = 8.8

Direction of taking of sample	E [MPa]	Rm [MPa]	A [%]
longitudinal	209 700	288.0	0.155
	206 174	315.3	0.217
transverse	193 973	339.0	0.243
	197 518	234.0	0.12
along	-	max. 400	25

Analysis indicates that formation of UFG is not effected by conventional discontinuous re-crystallisation, but by continuous re-crystallisation, characterised by consecutive division of very fine grains, and by migration of grain boundaries to short distance. Mechanical properties of the sample after deformation equal to 8.8 were verified by tensile test. The results are given in the table 2.

4. Conclusions

Tests made on the low carbon steel show a UFG structure. The obtained strength after severe deformation (e = 8.8) is very low. It is caused by big number of oxides in original dividing planes, which were formed at heating in oxidation atmosphere. In majority of cases the average size of grains, which occur also in the form of very fine lamellar structures, is around 0.5 µm. Next experiments have proved, that significantly finer grain is obtained that at lower temperatures. If there are created conditions for limitation of oxidation in dividing planes, it is possible to predict – subject to validity of relation given in Fig. 1 – the tensile strength twice or three times higher than the initial state of steel with conventional grain size.

Acknowledgements

The presented paper was realised within the frame of the project of the MPO, project FI-IM/033.

References

[1] V.M. Segal: Mat. Sci. and Eng., A197 (1995), p. 157.
[2] K. Morii, H. Mecking, G. Lutjering: Stability of the texture of Ti6Al4V during rolling in the two phases fields. Scripta Metallurgica, vol. 20, No. 12, p. 1795
[3] A. Hernas, G. Moskal, J. Dobrzanski: Graphite in the low-alloyed 15Mo3 steel after service. In Trans. VSB-TUO. XLVIII (2005), No.1, p. 97
[4] M. Greger, M. Widomská, A. Hernas, R. Kocich: Nano 04. VUT Brno 2004, poster
[5] M. Greger, R. Kocich: In 6th Scientific-technical conference Material in engineering practice´05. TU Žilina, Herľany 2005, p. 85.
[6] M. Dollár, A. Dollár: On the strength and ductility of nanocrystalline materials. J.Mat. Proc.Tech. 153 –158, 2004, p. 491-495.
[7] P. Kwapulinski, et al. Magnetic properties of amorphous and nanocrystalline alloys based on iron, J. Mat.Proc.Tech. 157-158 (2004), p. 735-742.
[8] S.M. Pytel, V. Tomaszewska-Gorecka, Odporność na pekanie ultraniskowegloewj stali bainitycznej. In AMME 2000, p. 443-448.
[9] T. Ungar, I. Alexandrov, M. Zehetbauer, Ultrafine-grained microstructures evolving during severe plastic deformation
[10] M. Furukawa, Z. Horita, T.G. Langdon, Conference: Strucure and Mechanical Properties of Nanophase Materials- theory and Computer Simulation vs. Experiment. (Materials Society Symposium Proceedings Vol. 634), Page: B8.5.1-B8.5.12Achieving superplasticity and super plastic forming through severe plastic deformation
[11] M. Furukawa, Z. Horita, M. Nemoto, T.G. Langdon, Conference: Ultrafine Grained Materials II. Proceedings of a Symposium, Page: 125-34,Factors influencing the development of ultrafine grained materials through severe plastic deformation
[12] R.K. Islamgaliev, R.Z. Valiev, Non-equilibrium grain boundaries in ultrafine-grained materials processed by severe plastic deformation
[13] Tsuji, Nobuhiro, Saito, Yoshihirolee, Seong-Hee;Minamino, Yoritoshi Advanced Engineering materials v 5 n 5 May 1 2003, ARB and other new techniques to produce bulk ultrafine grained materials
[14] Baikov Inst. of Metall./Mat. Science Russian Academy of Science, 119991 Moscow, Russian Federation Conference:Thermec 2003 Processing and Manufacturing of Advanced Materials, Madrid, Spain On the increase of thermal stability of ultrafine grained materials obtained by severe plastic deformation
[15] Watanabe, Hiroyuki; Mukai, Toshiji; Higashi, Kenji: Conference: Ultrafine Grained Materials II, Seattle, WA, USA Grain refinement and superplasticity in magnesium alloys.