

## STRUCTURAL STEELS WITH MICROMETER GRAIN SIZE: A SURVEY

### KONSTRUKCIJSKA JEKLA Z MIKROMETRSKIMI KRISTALNIMI ZRN: PREGLED

**Franc Vodopivec, Dimitrij Kmetič, Franc Tehovnik, Jelena Vojvodič-Tuma**

Institute of Metals and Technology, Lepi pot 11, 1000 Ljubljana, Slovenia  
franc.vodopivec@imt.si

*Prejem rokopisa – received: 2006-11-17; sprejem za objavo – accepted for publication: 2007-03-01*

Experimental findings and their theoretical interpretation related to the achieving of a  $\mu\text{m}$  grain size in structural steels with a microstructure of ferrite and pearlite are summarised. Several laboratory processing methods can be used to achieve this grain size. It seems that only DIFT (deformation induced ferrite transformation) offers the possibility of industrial use for thin sheets, while for thicker products DIFT can ensure the small grain size only for a thin surface layer. By very small grain size yield stress and tensile strength are increased, while elongation, reduction of area and strain hardening are decreased. For a grain size of 1.3  $\mu\text{m}$  upper shelf notch toughness is smaller, toughness transition temperature is lower and lower shelf notch toughness is higher than by the steel with the grain size of 6.8  $\mu\text{m}$ .

Key words: structural steels, ultrafine grain size, processing methods, effect of deformation and temperature, mechanical properties

Predstavljeni in interpretirani so eksperimentalni izsledki raziskovanja s ciljem doseganja mikrometrskih velikosti kristalnih zrn v konstrukcijskih jeklih z mikrostrukturo iz ferita in perlita. Tako velikost zrn je mogoče doseči z več metodami laboratorijskega procesiranja. Po dosedanjih spoznanjih je primerna za industrijsko uporabo za izdelavo tankih trakov le metoda DIFT (deformacijsko inducirana transformacija ferita). Po tej metodi je mogoče pri debelejših ploščah ustvariti zelo majhna zrna samo v tanki plasti ob površini. Pri zelo majhni velikosti zrn se povečata meja plastičnosti in trdnost, zmanjšajo pa se razteznost, kontrakcija in deformacijska utrditev. Pri jeklu z velikostjo zrn 1,3  $\mu\text{m}$  je v območju duktilnega loma zarezna žilavost manjša, prehodna temperatura žilavosti je tudi manjša, žilavost pod to temperaturo pa večja kot pri jeklu z velikostjo zrn 6,8  $\mu\text{m}$ .

Ključne besede: konstrukcijska jekla, zelo majhna kristalna zrna, metode procesiranja, vpliv deformacije in temperature, mehanske lastnosti

## 1 INTRODUCTION

Yield stress ( $R_E$ ) increases with decreasing grain size because the grain boundaries hinder the movement of dislocations produced by the cold deformation of metals according to the Hall-Petch equation:

$$R_E = R_0 + k \cdot d^{1/2} \quad (1)$$

With:  $R_0$  – constant depending on the chemical and phase composition of the steel and  $k$  – constant characteristic for the effect of linear grain size ( $d$ ).

For steels having an essentially ferritic microstructure the following relations were developed for the yield stress and for the notch toughness transition temperature<sup>1</sup>:

$$R_E / \text{MPa} = 104.1 + 32.6 w(\text{Mn}) + 84 w(\text{Si}) + 17.5 d^{-1/2} \quad (2)$$

$$ITT / ^\circ\text{C} = 19 + 44 w(\text{Si}) + 700 w(\text{N}) - 11.5 d^{-1/2} \quad (3)$$

By smaller grain size the temperature of cleavage of ferrite is lower and the yield stress greater. Other ferrite strengthening mechanisms increase also the temperature of cleavage fracture. For this reason, their exploitation for the increase of yield stress of structural steels is limited. It is predicted<sup>2</sup> that for a steel with a grain size

of 5  $\mu\text{m}$  the 50 % fracture appearance transition temperature of  $-100^\circ\text{C}$  would decrease to below  $-200^\circ\text{C}$  for a grain size of 1  $\mu\text{m}$ . The extent of the beneficial effect of grain size is shown in **Table 1** by comparison of the chemical composition and the share of other of strengthening mechanisms for three structural steels with a microstructure of ferrite and pearlite<sup>3</sup>. The industrial exploitation of the effect of grain size depends on the benefit obtained with the decrease of production costs which may result from the smaller content of alloying elements, the increased costs of the technology to achieve the aimed grain size resp. properties and the benefit of the use of the steel with increased yield strength for structures. In the last benefit several costs are included, f.i. lower transportation and erection costs for structures and a smaller quantity of welding consumables. Sometimes, the user can consider as important for the choice of steel also other criteria, f.i. the resistance of the steel to hydrogen embrittlement which is of essential importance for steels for vessels for liquid hydrocarbons. It is shown in **Figure 1** than steel C with a grain size of about 3  $\mu\text{m}$  conserves a much greater reduction of area after the NACE test of tensile test after a determined time of maintaining the specimens at stress level of 80 % of the yield stress in water saturated with  $\text{H}_2\text{S}$  than the

**Table 1:** Chemical composition and share of strengthening mechanisms for three structural steels

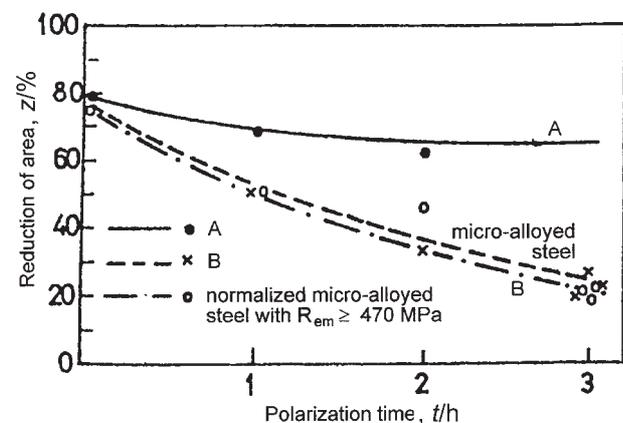
**Tabela 1:** Kemična sestava in masni delež mehanizmov utrditve za tri konstrukcijska jekla

Composition	Element in mass fractions w/%								
	C	Mn	Si	Al	Nb	Cr	Ni	Cu	Mo
Steel A	0.21	0.51	0.25	0.027	–	0.02	0.04	0.10	–
Steel B	0.17	1.32	0.32	0.009	–	0.21	0.13	–	–
Steel C	0.08	0.36	0.34	0.052	0.058	0.54	0.27	0.36	0.27
Share of yield stress increase, $R_E$ /MPa									
	SM 1	SM 2	SM 3	SM 4	SM 5	SM 6	SM 7	YS <sub>th</sub>	YS <sub>exp</sub>
Steel A	30	50	17	56	85	–	20	258	265
Steel B	30	61	17	104	135	–	28	372	377
Steel 3	30	15	17	136	254	9	43	504	522

SM 1 – ferrite yield stress, SM 2 – content of pearlite, SM 3 – Interstitial solution, SM 4 – Substitutional solution, SM 5 – Grain size, SM 6 – Dispersion, SM 7 – Precipitation in  $\gamma$  phase, YS<sub>th</sub> – calculated yield stress, YS<sub>exp</sub> – Experimental yield stress

steel B with a grain size of about 25  $\mu\text{m}$  inspite the by 1/3 greater yield stress of the first<sup>4</sup>. The fine grained steel in capable to retain in solution and in traps much more hydrogen than the coarse grained steel before the ductility is deteriorated to a significant extent. It is, thus, more resisting to hydrogen embrittlement and also more resistant to the delayed fracture due to the accumulation hydrogen at grain boundaries<sup>2</sup>.

It is shown later that there are processing routes which have the potential to obtain a grain size of 1  $\mu\text{m}$  in flat products with substantial thickness. These steel will find cost effective applications, but not major markets<sup>5</sup>. The reason is in the fact that the effect different major parameters affecting the grain size, f.i. steel chemistry, relation temperature – deformation intensity, cooling rate and transformation is not known to the extent allowing the industrial exploitation or the exploitation would require significant investments in the hot working technology, at least for products used for steel structures. Thus, by analysing the potential effect of grain size on structural steels, two aspects should be considered: the scientific and the technological – commercial.

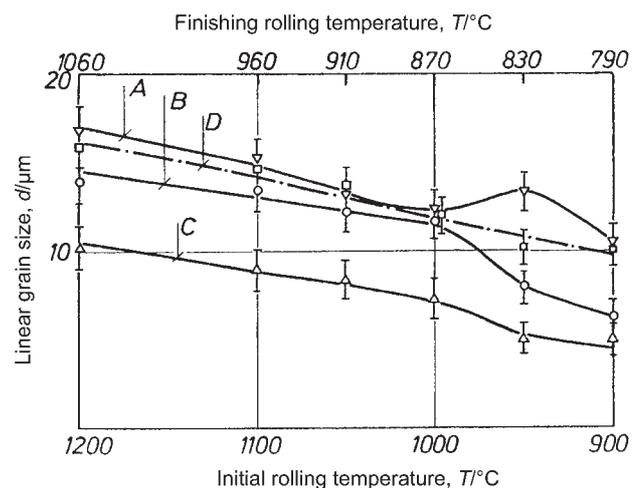


**Figure 1:** Results of the NACE test for the steels B and C in Table 1 and a microalloyed normalised steel with yield stress of 470 MPa<sup>4</sup>

**Slika 1:** Rezultati preizkusa NACE za jekla B in C iz table 1 in za mikrolegirano normalizirano jeklo z mejo plastičnosti 470 MPa<sup>4</sup>

By a given chemical composition, the parameters of hot working of steels of essential importance for the achieving of a small grain size are: the hot working temperature range, the finishing rolling temperature, the per pass deformation and the total plastic deformation in the range of temperature affecting the must the nucleation and the growth of recrystallised grains in deformed austenite or ferrite. One of the problems to overcome is the rate interpass growth of recrystallised grains and the rate of growth of ferrite grains after the austenite to ferrite transformation.

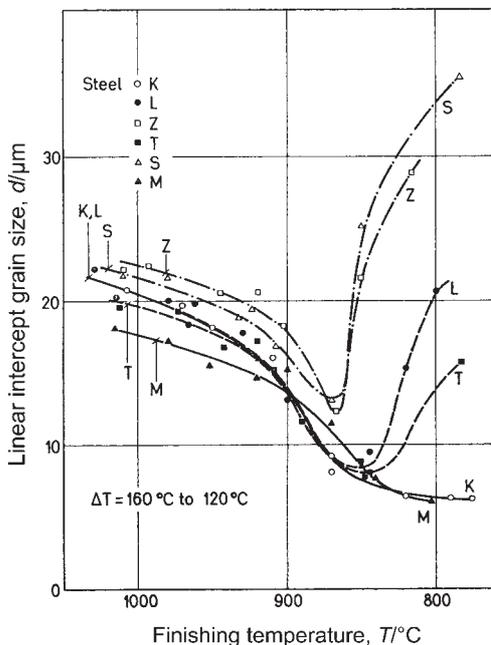
In Figure 2 the effect of rolling temperature range of 56 mm slabs to 12 mm plates in 6 passes temperature is shown for structural steels with different chemical composition<sup>6,7</sup>. All slabs were soaked at 1250 °C and cooled to a different initial rolling temperature. It is evident, that by several passes rolling the initial size of austenite grains does not affect the grain size in the



**Figure 2:** Effect of finishing rolling temperature on linear grain size for several structural steels rolled from 55 mm slabs in 6 passes to 16 mm plates and cooled in air on a warm bed

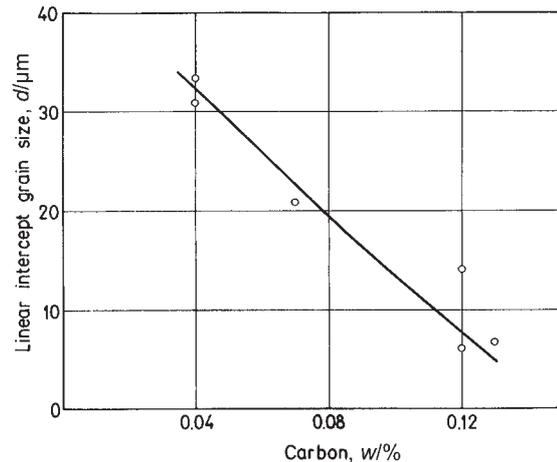
**Slika 2:** Vpliv temperature konca valjanja na linearno intercepcijsko dolžino za več konstrukcijskih jekel, ki so bila izvaljana iz 55-milimetrskih slabov v 16-milimetrske plošče v šestih vtikih in ohlajena na zraku na topli podlagi

rolled and air cooled steel. Also, in the applied rolling conditions, the effect of niobium carbide precipitation was very limited, since, only a slightly greater ferrite-pearlite grain size was obtained in steels with similar carbon content and without niobium although the virtually complete precipitation of niobium carbide during the rolling. In **Figure 3** the effect of finishing temperature on grain size is shown for steels with 0.04 % C to 0.13 % C<sup>8</sup>. The rolling regime was virtually identical to that for steels in **Figure 2**. At the same finishing temperature the grain size in the range of 7 μm to 33 μm was obtained. The shape of the curves for different steels on **Figure 3** shows that after a temperature, which depends on the content of carbon, coarser grain size is obtained at lower than by higher finishing rolling temperature. By a finishing temperature of 800 °C the grain size decreases virtually proportionally to the content of carbon in steel (**Figure 4**). From the shape of the relationship in **Figures 3 and 4** and considering the chemical composition of the steels, it was concluded that the difference between the theoretical transformation temperature of austenite and the real transformation temperature during the rolling was very small and that it depends mostly on the content of carbon in the steel. Also, **Figures 3 and 4** show that the growth of ferrite grains after the last pass (last partial deformation of about 20 %), is very fast. The strong effect of carbon on grain size in as rolled steels is explained with the increasing share of rolling performed in ferrite range



**Figure 3:** Effect of finishing rolling temperature on the ferrite pearlite grain size for steels with the content of carbon in the range of 0.04 % to 0.13 %. The tests were carried out in the same way as those in **Figure 2**

**Slika 3:** Vpliv konca temperature valjanja na velikost zrn ferita in perlita za jekla z ogljikom v razponu med 0,04 % in 0,12 %. Preizkusi so bili izvršeni na enak način kot pri jeklih na **sliki 2**



**Figure 4:** Relationship grain size versus carbon content for steels in **Figure 3** rolled in the temperature range from 900 °C to 790 °C

**Slika 4:** Velikost zrn v odvisnosti of vsebnosti ogljika za jekla s **slike 3**, ki so bila izvaljana v razponu temperature med 900 °C in 790 °C

with a per pass deformation of approximately 20 %, which is lower than that necessary for the static recrystallisation of this phase, which is of about 60 %<sup>9,10</sup>. According to<sup>11,12</sup> for the static recrystallisation of austenite only a per pass deformation of above 10 % is necessary. In absence of recrystallisation, the grain growth of ferrite, termed strain induced coarsening, occurs with a much greater rate than the growth of grains of ferrite produced with the transformation of recrystallised austenite at lower temperature. With the transformation of deformed and non recrystallised grains of austenite in Nb microalloying steel bainite grains of size more than one order of magnitude greater than the size of ferrite and pearlite grains in the surrounding matrix and formed by transformation of recrystallised austenite grains, were obtained, while the transformation of grains of deformed austenite in low carbon steel produced lenticular colonies of coarser, partially acicular ferrite and pearlite grains (**Figure 5**). It is clear, thus,



**Figure 5:** (magn. 200 times) Microstructure of the steel K from **Figure 3** rolled in the temperature range from 900 °C to 774 °C

**Slika 5:** (pov. 200-kratna) Mikrostruktura jekla K s **slike 3**, ki je bilo izvaljano v razponu temperature od 900 °C do 774 °C

that the achievement of  $\mu\text{m}$  and smaller size of ferrite grains in structural steels is a problem of rolling technology, and for a given chemical composition of the steel, it depends on the deformation and the temperature of final rolling passes, the austenite to ferrite transformation temperature and the cooling of the rolled steel. It is useful to remember that the general tendency in the development of modern structural steels is associated with a constant lowering of the content of carbon<sup>13</sup>, as shown in **Figure 6**.

In this survey the parameters related to the grain size of structural steels will be discussed considering the tests aimed to determine what can be achieved in laboratory and is, in this moment and in the near future, outside the potential of the present technology and what may be achieved with acceptable changes of the present technology.

**2 THEORETICAL ROUTES TO ACHIEVE A SMALL GRAIN SIZE**

The benefit of grain size is achieved with small crystal grains with high angle boundaries able to stop moving dislocations produced by cold deformation by testing of tensile properties at room temperature. High angle boundaries are achieved with recrystallisation of austenite and ferrite, mostly static, and with the austenite to ferrite transformation. It is, thus, logical to assume, than small grain size involving the phase transformation can be obtained only from austenite with very small grain size and the prevention of ferrite grain growth. If the small grain size is to be obtained with static recrystallisation of ferrite at hot rolling, a very great plastic deformation is necessary, in one pass or in several passes on condition of incomplete per pass relaxation of deformation energy.

The recrystallisation behaviour of austenite depends on the steel chemistry. For the calculations of the temperature of the end of static recrystallisation of austenite the following equation was deduced for the effect of different alloying elements in wt %<sup>14</sup>:

$$T_{nr} = 887 + 464 w(C) + 890 w(Ti) - 357 w(Si) + 6445 w(Nb) - 644 w(Nb)^{1/2} + 363 w(Al) \quad (3)$$

The effect of niobium is the greater and it is related to the precipitation of niobium carbide (NbC). For the calculation of the temperature of precipitation of this carbide the following relation was proposed<sup>14</sup>:

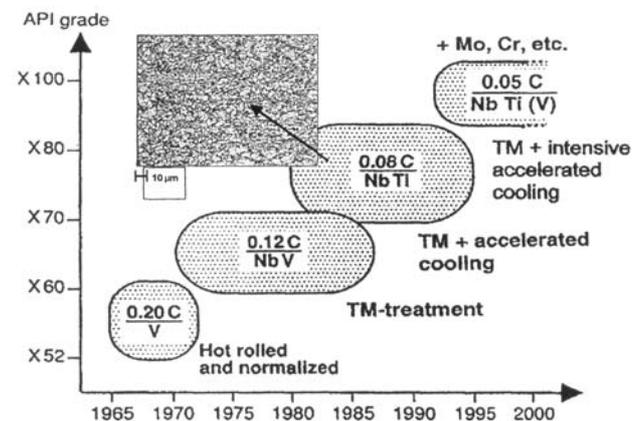
$$T_{NbC}/^{\circ}\text{C} = -6770 / \{ \lg [w(Nb) \times w(C)] - 2.26 \} - 273 \quad (4)$$

The rate of precipitation of NbC is at the same temperature for approximately three orders of magnitude greater during the deformation of austenite and for two orders of magnitude greater in deformed austenite<sup>15,16</sup> than in recrystallised austenite and the increase of strain rate lowers the recrystallisation and the precipitation temperature<sup>17</sup>. The explanation of the delaying effect of niobium is that strain induced precipitates hinder the

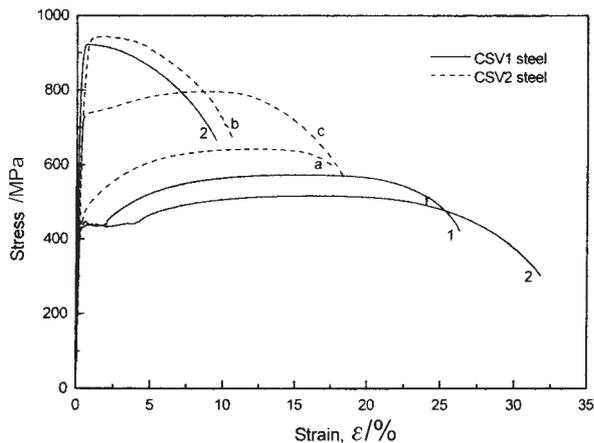
growth recrystallisation nuclei from reaching the critical size required for their growth in deformed austenite. Within this explanation it is not clear why the effect of titanium is smaller than that of niobium. By equal weight content, the atomic content of titanium is greater and the solubility product for titanium carbide is even smaller than that for NbC. For this reason, a similar effect of titanium would be expected even at higher temperature than that of niobium. It is interesting to note, with respect to the mechanism of the effect of niobium on recrystallisation of austenite, that it was found with dilatometric investigations of recrystallisation and precipitation, that niobium in solid solution in ferrite delayed the recrystallisation of this phase<sup>14</sup>. Other alloying elements have a much smaller effect of the temperature of static recrystallisation of austenite and, for this reason, can not be, exploited for the achieving of small grain size. In all cases, the presence of a determined number of precipitates is necessary to prevent the growth of recrystallised austenite and ferrite grains.

The microstructure of fine grained structural steels consists of ferrite, pearlite and bainite in different combinations and can be achieved with the transformation of fine grained austenite. For a given steel chemistry, the austenite grain size depends on the extent of deformation and the growth of recrystallised grain in interpass time, the time of cooling below the minimal grain growth temperature or the time to the transformation to ferrite. It was found that, independently on the cooling rate, after holding of the deformed austenite for 100 s at 900 °C, the steel had a ferrite grain size 1  $\mu\text{m}$  greater than without holding time<sup>14</sup>. The explanation was in the rapid coarsening of NbC precipitates.

In laboratory, it is possible to vary in a large range all the parameters affecting the grain size. At this time, the smallest grain size is achieved with ECAP<sup>18</sup> (equal channel angular pressing) or hot pressing. The deformation energy is dispersed mostly as heat, for this reason, test with great deformation or deformation rate are not isothermal. The rate of plastic deformation is generally



**Figure 6:** Evolutionary trend for high strength steels for linepipes  
**Slika 6:** Smer evolucije visokotrdnih jekel za cevi



**Figure 7:** Stress-strain curves for two steels before V(1) and after ECAP V(2)

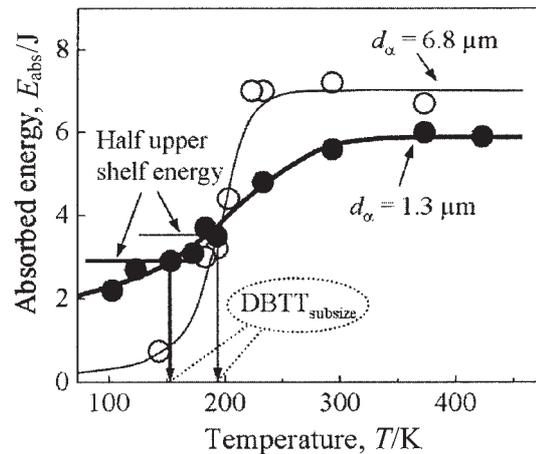
**Slika 7:** Odvisnosti napetost-deformacija za dve jekli pred ECAP V(1) in po njem V(2)

high and, for this reason, the deformation energy may be generated adiabatically and the steel in the deformation zone heated significantly above the nominal deformation temperature.

With ECAP processing<sup>18</sup> a grain size below 1  $\mu\text{m}$  can be obtained with interstitial free steels. With 4 passes of ECAP at 623 K a ferrite grain size of 0.3  $\mu\text{m}$  and severely deformed pearlitic cementite were achieved in a steel with 0.15 % C, 0.25 % Si and 1.1 % Mn and the initial intercept length of  $\sim 30 \mu\text{m}$ . The yield stress was increased from 300 MPa to 944 MPa, while, the ductility was decreased to less than one half of the initial level. The addition of 0.06 % V to the steel had no visible effect on the properties after ECAP inspite of the fact, that the initial grain size was of  $\sim 10 \mu\text{m}$ , thus only 1/3 of that in the case of the vanadium free steel. The increase of ECAP passes from 4 to 8 did not affect the grain size and the yield stress, it increased however the misorientation of the grain boundaries<sup>19,20</sup>. The conclusions were that that by multiple ECAP the effect of initial grain size is not significant.

The tensile properties were changed significantly after ECAP deformation of the 0.15 % C, 0.25 % Si, 1.12 % Mn, 0.34 % V and 0.012 % N, yield stress was increased from 435 MPa to 920 MPa, tensile strength for 568 MPa to 920 MPa, uniform elongation was decreased from 17 % to 2 % and total elongation from 28 % to 9 % (**Figure 7**)<sup>21</sup>. If during the ECAP test nanosize precipitation of vanadium carbide occurred, the precipitates improved the thermal stability of nanostructure and of tensile properties of the steel, while, the particles obtained with normalisation before ECAP had no significant effect of the thermal stability of the ECAP nanostructure<sup>21</sup>.

With ECAP at 500 °C and intercritical annealing of the 0.15 % C, 0.25 % Si and 1.1 % Mn the achieved grain size of ferrite grains and martensite islands was of 0.8  $\mu\text{m}$ <sup>22</sup>, the yield stress, of 540 MPa, tensile strength of 890



**Figure 8:** Effect of test temperature on absorbed energy for two steels with grain size 6.8  $\mu\text{m}$  and 1.3  $\mu\text{m}$

**Slika 8:** Vpliv temperature preizkusa na energijo preloma za dve jekli z velikostjo zrn 6,8  $\mu\text{m}$  in 1,3  $\mu\text{m}$

MPa, uniform elongation of 9.8 % and total elongation of 17.6 %. By difference of results achieved with other types of steel, the investigated steel exhibited an extensive strain hardening.

With four pass compression with a total strain of  $\varepsilon = 1.6$  in the temperature range from 1143 °C to 823 °C the grain size of a 0.22 % C and 0.74 % Mn steel was diminished from 6.8  $\mu\text{m}$  to 1.3  $\mu\text{m}$ , yield stress increased from 360 MPa to 540 MPa<sup>23</sup> and the elongation was decreased from 31 % to 16 %. This decrease was explained in terms of smaller strain hardening due to the spreading of dislocation in the grain boundaries of ferrite grains. Of special interest in the finding that by small grain size the upper shelf absorbed energy was smaller than by coarse grain size, while the transition temperature was lower and the transition range was greater (**Figure 8**). The greater notch toughness below the value of half upper shelf energy was explained in terms of delamination occurring at the fracturing in lower temperature range, which may be related to a stronger texture of crystal grains in the fracture plane.

The one pass hot pressing eliminates the changes of micro/nano structure during the interpass time, avoids the multi axial deformation and enables the analysis of the micro/nano structure obtained at different strain level<sup>24</sup>. The transformation grain refinement (TGR) and recrystallisation grain refinement (RGR) are suggested as potential routes to achieve  $\mu\text{m}$  grain size. Both routes can be carried out f.i., with tests of ECAP, hot pressing or warm rolling. By one pass pressing of 0.16 % C, 0.41 % Si and 1.43 % Mn at 823 °C and 10<sup>-1</sup>s they were able to distinguish the different steps of the formation of small grains. First the original grain are compressed and elongated in the direction of metal flow and low angle boundaries are introduced because of the parallel rotation of elongated grains. At the strain of 2.5 fine equiaxed grains appear at high angle boundaries of initial

grains, at the strain of 4 the share of fine new grains is significant and it increases continuously with the increasing plastic deformation. The formation of these grains is explained with work hardening accompanied with grain subdivision and dynamical recovery occurring simultaneously at warm working temperatures. With warm pressing deformation in four passes with the per pass deformation of  $\varepsilon = 0.4$  in temperature range from 600 °C to 710 °C in a 0.36 % C, 0.53 % Mn, 0.22 % Si equiaxed ferrite grains of size 1  $\mu\text{m}$  to 2  $\mu\text{m}$  and an uniform distribution of even smaller cementite particles were attained<sup>25</sup>.

The different routes to achieve small grain size are discussed also in<sup>26</sup> where also experimental work was performed with the aim to verify the exploitation of the deformation induced ferrite transformation (DIFT) and the accumulative roll bonding. The investigation was performed in the frame of a ECSC project aimed to identify whether ultrafine grained steels were a potential commercial opportunity or just an academic curiosity. It was established that deformation induced ferrite transformation (DIFT) occurs at a critical strain, which is related to the chemistry of the steel and that carbon in solution in austenite increases the critical strain and retards the DIFT.

The process of DIFT consists of rolling a coarse grained steel close to the  $A_{r1}$  level with a reduction of 30–40 %<sup>27</sup>. The undecooling due to the roll chilling and the high shear strain at the sheet surface, increases greatly the nucleation rate in austenite grains. In this way, a very rapid austenite transformation is achieved over the whole austenite grain. It seems that the strain energy of deformed austenite may induce the transformation to ferrite slightly above the  $A_{r3}$  point<sup>28,29,30</sup>, in agreement with **Figure 3**.

In the thermodynamic analysis of DIFT<sup>31</sup> it is assumed that the deformation elongates austenite grains, increases the grain boundary area and enhances the disorder of the grain boundary structure and the grain boundary energy per unit area. Analytical relations were developed to calculate the increase of free energy in dependence of the deformation degree and rate. Two nucleation sites for the transformation induced ferrite (DIF) were observed: austenite grain boundaries and two sites in the interior of austenite grains: deformation bands and the new formed  $\gamma/\alpha$  interface. The share of DIFT in the steels increases strongly with the deformation and it is of approximately 25 % by a strain of 0.4 and of 80 % by a strain of 1.2. With laboratory DIFT rolling of a 0.09 %C, 0.47 %Si, 1.38 %Mn, 0.1 %V, 0.04 %Nb, 0.02 %Al and 0.018 %N steel in the temperature range of 1473 K to 1093 K with a strain of 0.93 at the final three passes grain size of 1.5  $\mu\text{m}$  was obtained.

With DIFT in a microalloyed structural low carbon steel<sup>32</sup> (X65 steel) a grain size of 1.22  $\mu\text{m}$  and the volume share of DIFT ferrite of 71 % were obtained by the hot rolling reduction of 69 %, the grain size of 0.92

$\mu\text{m}$  and a volume share of DIFT ferrite of 98 % were obtained after a deformation of 88 %. The precipitation of niobium should occur before the final rolling passes. If not achieved, coarser grain size, similar to that in **Figure 5**, is obtained after air cooling.

The 0.08 %C, 1.74 %Mn and 0.18 %Ti steel was rolled in 6 passes from 30 mm to 5 mm with the finishing temperature in the range 790 °C to 820 °C and cooled with the rate in the range of 20 °C/s to 40 °C/s to the coiling temperature of 550 °C<sup>33</sup>. A microstructure consisting of ferrite of average linear size of approximately 1.5  $\mu\text{m}$  by a cooling velocity of 20 °C/s and bainite by a cooling rate of 30 °C/s were obtained. With the microstructure of ferrite the yield stress of 500 MPa and the elongation of 27 % were obtained, while with a microstructure of 90 % of bainite the yield stress was of 835 MPa and the elongation of 20 %.

The ferrite grain size of approximately 2  $\mu\text{m}$  seems to be the minimal size, which can be obtained by the DIFT mechanism and heavy rolling deformation of supercooled austenite using the existing rolling facilities<sup>34</sup>. Further refining doesn't seem to improve the mechanical properties as compared to the effects to be made. In this reference three temperature ( $T_d$ ) domains of grain refinement by heavy deformation are mentioned. Deformation of supercooled austenite ( $T_d > A_{r3}$ ) and DIFT refinement mechanism and limit grain size of 2  $\mu\text{m}$ , deformation of a duplex austenite + ferrite initial microstructure ( $A_{r3} > T_d > A_{r1}$ ) and refining by DIFT or DRX (dynamic recrystallisation of ferrite) and limit grain size of 1  $\mu\text{m}$  and deformation of ferrite ( $T_d < A_{r1}$ ) with the limit grain size of 0.6  $\mu\text{m}$ . Ferrite grain size obtained with dynamic transformation is finer than that obtained with static recrystallisation. No data on the extent of deformation are given in this reference.

### 3 CONCLUSION

Micrometer grain size can be obtained for low carbon structural steels with several methods. Virtually all of them are suited for laboratory processing, only DIFT (deformation induced ferrite transformation) and low temperature transformation of recrystallised austenite seems to offer the industrial feasibility. Several authors confirm that by  $\mu\text{m}$  grain size yield stress and tensile strength are greatly increased, while elongation is significantly decreased. In one reference it was found, that by the upper shelf ductile fracture energy was lower for the same steel by the 1.3  $\mu\text{m}$  than by the 6.8  $\mu\text{m}$  grain size. On the contrary below the temperature of half of the maximal fracture energy, the toughness was greater for the steel with smaller grain size.

The DIFT processing seems to be suited for the production of thin sheets, for thicker product the  $\mu\text{m}$  size of grains can be obtained only in a relatively thin surface layer. For thicker plates, a small grain size can be achieved with strictly controlled thermomechanical

rolling involving the completed recrystallisation of plastically deformed austenite, its transformation to polygonal ferrite at a low temperature, and a sufficiently rapid cooling to prevent the ferrite grain growth. For such processing only niobium microalloyed steel are suited. The chemical composition of the steel should ensure a low temperature of precipitation of niobium carbide and a low transformation temperature of recrystallised austenite to polygonal ferrite.

#### 4 REFERENCES

- <sup>1</sup> F. B. Pickering, T. Gladman: Metallurgical Development in carbon steels; ISI, 1963, 10–20
- <sup>2</sup> Ultra Steels for the 21<sup>st</sup> Century; Center for Structural Materials Research, National Institute for Materials Research, Japan, 2001
- <sup>3</sup> F. Vodopivec, J. Vojvodič - Tuma, M. Lovrečič - Saražin: Kovine Zlit. Tehnol. 32 (1998), 463
- <sup>4</sup> S. Ažman, L. Vehovar, J. Vojvodič-Tuma: Razvoj čelika mikrolegiranog niobijem i molibdenom (Development of a steel microalloyed with niobium and molybdenum), Zavarivanje 96, Beograd, 58–60
- <sup>5</sup> J. A. A. Hove: Mat. Sci. Techn. 16 (2000), 1264
- <sup>6</sup> F. Vodopivec, M. Gabrovšek, J. Žvokelj: Želez. zb. 17 (1985), 17
- <sup>7</sup> A. Preloščan, F. Vodopivec, I. Mamuzić: Mater. tehnol. 36 (2002), 181
- <sup>8</sup> F. Vodopivec, M. Gabrovšek, J. Žvokelj: Transactions ISIJ 28 (1988) 117
- <sup>9</sup> D. N. Hawkins: Metals Techn. 5 (1978), 37
- <sup>10</sup> S. Gohda, T. Watanebe, J. Hashimoto: Trans. ISIJ 21 (1981), 6
- <sup>11</sup> T. Tanaka, N. Tabata, T. Hatamura, C. Shiga: Microalloying 75, UCC Corpor., New York, 1975, 107
- <sup>12</sup> I. Kozasu, C. Ouchi, T. Sampei, T. Okita: Microalloying 75, UCC Corpor., New York, 1975, 120
- <sup>13</sup> G. Buzzichelli, E. Anelli: ISIJ Intern. 42 (2002), 1354
- <sup>14</sup> C. Mesplont: Grain refinement and high precipitation hardening by combining microalloying and ultra fast cooling; Proceedings of the 1<sup>th</sup> International Conference on super – high strength steels, Rome, Italy, 2–4 Nov. 2005
- <sup>15</sup> I. Weiss, J. J. Jonas: Metall. Trans. 11 A (1980), 403
- <sup>16</sup> J. J. Jonas, I. Weiss: Met. Sci. 3 (1979), 238
- <sup>17</sup> S. F. Medina, A. Quispe: Mat. Sci. Techn 16 (2000), 635
- <sup>18</sup> Z. Horita, M. Furukawa, M. Nemoto, T. G. Langdon: Mat. Sci. Techn. 16 (2000), 1239
- <sup>19</sup> D. H. Shin, J. J. Park, S. Y. Chang, Y. K. Lee, K. T. Park: ISIJ Intern. 42 (2002), 1490
- <sup>20</sup> D. H. Shin, W. G. Kim, J. Y. Ahn, K. T. Park, N. J. Kim: Fabrication and tensile properties of ultrafine grained steels; Proceedings of the 1<sup>th</sup> International Conference on super – high strength steels, Rome, Italy, 2–4 Nov., 2005
- <sup>21</sup> K. T. Park, S. Y. Han, D. H. Shin, Y. K. Lee, K. S. Lee: ISIJ Intern. 4 (2006), 1057
- <sup>22</sup> K. T. Park, Y. K. Lee, D. H. Shin: I/ISIJ Intern. 45 (2005) 9, 750
- <sup>23</sup> R. Song, D. Ponge, D. Raabe: Mechanical properties of an ultrafine grained C-Mn steel; Proceedings of the 1<sup>th</sup> International Conference on super – high strength steels, Rome, Italy, 2–4 Nov., 2005
- <sup>24</sup> S. V. S. Narayana Murty, S. Torizuka, K. Nagai: ISIJ Intern. 45 (2005), 1651
- <sup>25</sup> L. Sorojeva, R. Kaspar, D. Ponge: ISIJ Intern. 44 (2004), 1211
- <sup>26</sup> I. Salvatori: Ultrafine grained steels by advanced thermomechanical processes and severe plastic deformation; Proceedings of the 1<sup>th</sup> International Conference on super – high strength steels, Rome, Italy, 2–4 Nov., 2005
- <sup>27</sup> P. J. Hurley, B. C. Module, P. D. Hogson in Thermomechanical Processing of steels 2000, London, Mai 2000, 476 (loc. cit. ref. 24)
- <sup>28</sup> M. R. Higson, P. D. Hogson: Mater. Sci. Technol. 15 (2000), 85
- <sup>29</sup> J. K. Choi, D. H. Seo, K. Umk, W. Y. Choo: ISIJ Intern. 43 (2003), 764
- <sup>30</sup> S. C. Hong, S. H. Lim, H. S. Hong, K. J. Lee, D. H. Shin, K. S. Lee: Mat. Sci. Techn. 20 (2004), 207
- <sup>31</sup> X. Sun, Q. Liu, H. Dong: Deformation induced ferrite transformation and grain refinement in low carbon steel; Proceedings of the 1<sup>th</sup> International conference on Super – high strength steels, Rome, Italy, 2–4 Nov. 2005
- <sup>32</sup> H. Dong, Y. Gan, Y. Weng: Research activities on advanced steels in Nercast; Proceedings of the 1<sup>th</sup> International conference on Super – high strength steels, Rome, Italy, 2–4 Nov., 2005
- <sup>33</sup> J. K. Choi: Development of high strength and high performance steels at Posco through Hipers-21 project; Proceedings of the 1<sup>th</sup> International conference on Super – high strength steels, Rome, Italy, 2–4 Nov. 2005
- <sup>34</sup> H. L. Yi, L. X. Du, G. D. Wang, X. H. Liu: ISJ Intern. 46 (2006), 754