Achieving ultra-fine grained microalloyed steel by severe plastic process

G Gurau¹, C Gurau¹*, L Gurau²

¹Faculty of Engineering”Dunărea de Jos” University of Galati, Domnească Street, 47, RO-800008, Galati, Romania
²ArcelorMittal Europe Flat Products - Business Division East, Dabrowa Gornicza - Poland

* carmela.gurau@ugal.ro

Abstract. Latterly, it is of interest to enhance combination of strength and toughness of steel for advanced products, therefore the objective of this research is to improve the mechanical properties of high strength low - or ultra - low alloy (HSLA) steels through grain refinement. The ultra-fine grained S420NL steel were produced by high speed high pressure torsion (HSHPT) at room temperature. This severe plastic deformation method was chosen because it is one of the most feasible and rapid ways for nanostructuring various metallic materials down to ultrafine grain range. The procedure key parameters of deformation are described in detail in the paper. The influence of severe plastic deformation applied in cast state steel on microstructure changes and deformation behaviour was investigated. Optical microscopy, SEM and energy dispersive x-ray spectroscopy (EDX) analysis carried out on the samples revealed that severe plastic deformation led to the subsequent formation of an ultrafine-grained microstructure. Sample hardness increased rapidly with progress of torsion deformation degree.

1. Introduction

Since the discovery the concept of nanocrystalline materials (H. Gleiter 1989) [1] that offer excellent mechanical properties, good corrosion resistance as well as good weldability and so on, severe plastic deformation (SPD) has been developed as attractive method to refine the structure grains metallic materials to the ultrafine scale or even under 100 nm. Ultrafine grained or nanograined materials produced by HPT demonstrated extremely high strength and high hardness [2-6]. Recently, bulk ultrafine-grained (< 1 μm) tempered martensitic 9–12% chromium steels steel was processed by high pressure torsion (HPT) severe plastic deformation (SPD) method [7] to exhibit a faster minimum creep rate and higher ductility in comparison with coarse-grained alloy ductility. Withal high strength low-carbon steels for small modular reactors (SMRs) was designed by HPT [8]. Our steps proceed very much in the same way of refines the structure of low-carbon steel based on severe plastic deformation by high speed high pressure torsion (HSHPT) [9]. HSHPT consists in simultaneous induce of shear strain, pressure and friction sample-anvils by high speed rotation of superior piston. This severe plastic deformation method was chosen because it is one of the most feasible and rapid ways for nanostructuring various metallic materials down to ultrafine grain range. The contribution of this paper is to provide the effect of different degree of deformation on severely plastic deformed S420NL steel.
2. Experimental

It was carrying out a process of severe deformation by HSHPT. High speed high pressure torsion is a very efficient method which refines the structure of metallic materials to the ultrafine (< 1 µm) or even the nanometer (< 100 nm) scale [9-12]. In this study a ferritic steel with traces of pearlite grains was in use and its composition is presented in Table 1. From the middle of the steel rings with outer diameter of 20 mm, inner diameter of 10 with a thickness of 2÷3 mm were machined from transversal direction of the sheet. These rings were processed in tronconic elements by HSHPT using an initial pressure of 25bars, nominal pressure between 0.06GPa to 1.15GPa, a rotational speed of 900r/min. The degree of deformation imposed onto the HSHPT-rings was calculated with: $\varepsilon = \ln(h_i/h_f)$. The logarithmic strain levels achieved were from 0.1 up to 2.04. The thinnest truncated cone shell exhibited a thickness of 0.55mm. More details to the used HSHPT-setup can be found elsewhere [10].

| C   | Mn | Si | S   | P   | Al  | N   | Cu  | Ni  | Cr  | Mo  | Nb  | Ti  | V   |
|-----|----|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] | [%] |
| 0.16 | 1.60 | 0.50 | 0.005 | 0.015 | 0.04 | 0.015 | 0.140 | 0.200 | 0.070 | 0.010 | 0.120 |

To determine the influence of HSHPT severe plastic deformation a series of experimental tests were conducted to evaluate the structure and mechanical properties. A detail analysis involving macroscopic feature and microstructure analysis of the microalloyed steel was also performed by optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The examination was carried out using an OLYMPUS BX51 microscope and a ZEISS microscope, respectively. Prior to microstructural examination the samples were prepared by standard metallography techniques. The hardness measurements were carried out on a Vickers hardness tester with a load of 100 gf and a abide time of 15 s. For each data point 6 indents were made at equivalent geometrical positions and averaged.

![Figure 1](image-url)
3. Results and discussion

Latterly, it is of interest to enhance combination of strength and toughness of steel for advanced products [12-13], therefor the objective of this research is to improve the mechanical properties of high strength low - or ultra - low alloy (HSLA) steels through grain refinement. HSHPT can impose a shear strain far beyond the strain level attainable by conventional plastic deformation methods such as rolling or extrusion. The key parameters of severe plastic deformation are described above in this paper. Figure 1 shows the major parameters of severe plastic deformation in sample that bear 1.01 logarithmic degree. The plastic deformation lasted less than 4s. The rotational superior anvil slightly decreased during deformation. The torque attained was 100Nm. The maximum pressure required in process was only 0.17GPa. The temperature of sample reach 550°C on the strength of friction driven by high speed rotation of superior punch.

The macrostructure analysis were done through SEM and energy dispersive X-ray spectroscopy (EDX) (Figure 2). The as-received sample has a large non-metallic compound made of MnS and Al, Ca and Si oxides formed by the dezoxidizing step (Figure 2a). In severe plastic deformed elements the nonmetallic inclusions is splitting and shrinking as can be seen from Figure 2(b and c).

![Figure 2](image_url)

**Figure 2.** Comparison of the nonmetallic inclusions in S420NL steel: as-received (a), after HSHPT with $\varepsilon=1.87$ (b) and $\varepsilon=2.04$ (c).

| Sample a | C-K   | O-K   | Mg-K  | Al-K  | Si-K  | S-K   | Ca-K  | Ti-K  | Mn-K  | Fe-K   | Ni-K  |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|-------|
| $pt1$    | 2.58  | 3.33  | 28.39 | 35.30 | 0.08  | 20.05 | 10.26 | 0.01  |       |        |       |
| $pt2$    | 1.31  | 36.77 | 2.71  | 42.59 | 1.01  | 5.65  | 1.04  | 1.03  | 7.84  | 0.06   |       |
| $pt3$    | 1.12  | 0.40  |       |       |       |       |       |       |       |        | 96.59 |

| Sample b | C-K   | O-K   | Si-K  | Mn-K  | Fe-K  |
|----------|-------|-------|-------|-------|-------|
| $pt1$    | 4.05  | 1.08  | 22.93 | 1.13  | 70.81 |
| $pt2$    | 0.98  | 0.34  | 1.92  | 96.76 |       |

| Sample c | C-K   | O-K   | Mg-K  | Al-K  | Si-K  | S-K   | Ca-K  | Ti-K  | Mn-K  | Fe-K   |
|----------|-------|-------|-------|-------|-------|-------|-------|-------|-------|--------|
| $pt1$    | 0.95  | 5.76  | 1.48  | 13.46 | 0.18  | 6.99  | 5.63  | 0.72  | 5.72  | 59.11  |
| $pt2$    | 1.21  | 0.07  | 4.26  | 0.46  | 4.72  | 4.27  |       |       |       |        |

The microstructure of the as-received condition has coarse ferrite colonies (bright) and few strings of pearlite phase (dark) as shown in Figure 3. The low carbon steel in rolling state consists on large volume of ferrite grains. The size of ferrite grains vary quite a bit between 4 µm and 20 µm. The volume fraction of the fine pearlite phase is observed not more than 3vol%. The strings pearlite are arranged in linear arrays, surrounded by the ferrite grains.

The alloy exhibit a process of refining the initial ferrite grains more pronounced as the application an increased degree of deformation (Figure 4a-f).

The curled and wavy morphology of the ferrite with decreased size and elongated grains confirms that the ring has deformed plastically (Figure 4a).
Figure 3. OM micrographs illustrate the phase volume fraction (a) and the size of the real ferrite grain (b) of the as-received samples. At higher logarithmic degree of deformation, the ferritic structure becomes gradually aligned parallel to the shear direction.

Figure 4. OM images showing the characteristic microstructures of incremental strain after HSHPT
Moreover, there is also a recognizable refinement and substructure formation within the ferrite phase (Figure 4b-d). The type, morphology, size, and density of precipitates significantly affect the overall properties of coarse grained and submicron materials [7]. Precipitates inside initial rings and tronconic elements after severe plastic deformation can be intermetallic or ceramic materials. The type of these has been described in above EDX investigation. Most of the secondary phases dispersed within and at grain boundaries of ferrite phase matrix consist on cementite, intermetallic compound. These precipitates suffers a fragmentation and distribution process along the sliding lines, as can be observed better at low deformation degrees. Besides grain refinement and fragmentation of precipitates, SPD can also induce diffusive phase transformation. This process occurs by shearing strength subsequent under high pressure concomitantly with rotating. However, the applied deformation induces strain to deform the ferrite matrix and enlarge the volume of pearlite. The increased volume fraction of fine lamellas and globular pearlite concur to the enhancing mechanical strength. The pearlite manifest higher strength because of cementite and this contributes to superior mechanical properties. The fine curved strings of pearlite are distributed and diffused in the grain boundaries of ferrite zone as shown in Figure 4e. As the shear strain increased (ε=1.3) the representative microstructural feature of the steel was uniformly distributed equiaxed submicron grains, as illustrated in Figure 4f. The bright grains under 1µm are ferrite phase surrounded by a very fine and uniformly distributed perlite.

The homogeneity of the microstructure increases with an increasing value of the plastic strain occurring during HSHPT.

The increased pearlite matrix with fine and globular morphology contributes towards improving the mechanical properties of the HSHPT’ed processed sample. In this type of processing, the sample are heated by friction inside mold. The high speed rotation of anvil (900rpm) on the initial pressed ring with 25 bars drives heating. However, a complex plastic deformation at temperature occurs. At above 500°C torsion and pressing takes place simultaneously. In this moment the high pressure is applied on sample. High pressure of the order of 1GPa lead to refining the structure by concomitant shearing and sliding of crystalline grains in compact blocks.

It is well known that dislocation slip and deformation twinning are the deformation mechanisms in coarse grained metallic materials. The same two mechanisms which induced grain refinement processes take place during the SPD. However, deformation twinning has been rare in BCC materials SPD-processed and these only in case of induced by shock. The twinning process is specific to low temperature deformation. In case of HSHPT the samples are heated before the nominal pressure is applied. In the ferritic BCC phase the major deformation mode were found as dislocation activities like dislocation multiplication, accumulation, and annihilation via the formation of grain boundaries [14]. The dislocation structures are also affected by the stacking faults energy (SFE) of the materials.

![Figure 5. OM images showing the features of grain refinement and preferential slip directions on sample deformed with ε=0.29 (a) and ε=1.11 (b).](image)

During the HSHPT more than in other SPD induced continuous grain growth process from a much smaller grain size to the final steady-state grain size which remains unchanged with further straining
The average steady-state grain size depends on material properties and more to the SPD technique and processing parameters (applied pressure, strain rate and temperature reached during deformation).

The microstructural evolution of this HSLA steel having different degree of deformation has been highlighted in Figure 6, by SEM investigation. The SEM of the sample after deformation with $\varepsilon=1.87$ showed the formation of annular and waved flow curves through submicron grains (Figure 6a). SEM analysis carried out on the sample incorporating 2.04 deformation degree revealed that severe plastic deformation led to the subsequent formation of a significant refinement (Figure 6b).

![Figure 6](image_url)

**Figure 6.** SEM micrographs of HSHPT’ed samples after deformation with: $\varepsilon=1.87$ (a) and $\varepsilon=2.04$ (b)

The individual grains or grains boundaries were outside the range of detection by standard SEM observation.

![Figure 7](image_url)

**Figure 7.** Vickers's microhardness evolution of HSHPT’ed samples with respect to the logarithmic degree of deformation

This method applied on bulk metallic HSLA steel manage to achieve ultrafine grained structure that possess mechanical properties that expand rolling sheet performance. The microstructural evolution, morphological changes in ferrite and pearlite colony and grain refinement of the deformed samples were correlated with the mechanical properties. In order to understand the advantage of this process, the HSHPT’ed samples were compared with the initial hot-rolled sample and the results are presented in Figure 7. The hardness of samples increased rapidly with progress of torsion deformation degree. The
Vickers hardness increases four times. The experimental tests and analysis results show that there are significant improved in the material properties of severe deformed coned rings when comparing it with the thick steel plates Standard, EN10027-1 and CR10260.

4. Conclusion

We have assessed the potential improvement of the mechanical characteristics in the S420NL low alloy (HSLA) steel by high speed high pressure torsion (HSHPT). The ultra-fine grained S420NL steel were produced by HSHPT at room temperature. Microstructure investigations show that this SPD method significantly changes the shape, distribution and grains size. The microstructure essentially refined down to the submicron level. As the shear strain increased at $\varepsilon=1.3$ the representative microstructural feature of the steel was uniformly distributed equiaxed submicron grains of ferrite phase surrounded by a very fine and uniformly distributed perlite. Beyond $\varepsilon=2$ individual grains or grains boundaries were outside the range of detection by standard SEM observation. The HSHPT-produced UFG structured S420NL steel demonstrates high hardness (587 HV). The Vickers hardness increases four times as against thick steel plates. Results obtained by this method allow further research to obtain low-weight S420NL products nanostructured by HSHPT with much improved properties.

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