### Mechanical and Structural Changes of Austenitic Sheet Metal Alloy during Biaxial Tensile Straining by Hydraulic Bulging

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Abstract: Austenitic stainless steel sheets are important technological materials due to their diverse applications ranging from chemical transportation, shipbuilding, automotive and aircraft industry. During deformation at room temperature or at work the sheet materials undergo phase, structural and mechanical changes. As the deformation increases, these particular changes may become critical for occurrence of local weak points and consequent breakage.

In order to determine the phase, structural and mechanical changes of sheet materials, experimental arrangements allowing deformation greater than 30% are increasingly used. Such a method of greater use is the hydraulic bulding. As the biaxial tensile deformation increases, the indicator diagrams display apparent serrated flow and a change in the magnetic permeability. The slow rate strain transformation under biaxial tensile strength is revealed by means of microstructural, XRD analysis and hardness tests. The correlation between the mechanical and structural response of the different zones of the deformed sheet material is revealed.

Key words: Stainless steel sheets; Biaxial Tensile Strength; Austenite; Martensite transformation, Microstructure, Hardness, XRD

#### 1. INTRODUCTION

The metastable austenitic alloy is susceptible to solid-state transformation via displacement of atomic planes under plastic deformation at low temperatures. Recent studies show that there is a gap of theoretical models from experimental results for the mechanical properties of these sheet materials in degrees of deformation greater than 30% in the uniaxial tension [1, 2, 3]. The most popular test methods for increasing the deformation values and determining the mechanical properties of the materials are based on the scheme of hydraulic bulging [4, 5]. The latter provides simple experimental test unit, which is easily serviced and the up to date measuring equipment allows to achieve sufficient accuracy. The hydraulic bulging method closely represents another technological method of deformation of sheet alloys called "hydro-forming" [2]. This test scheme makes it possible to determine the levels of extension of the materials in production conditions. By combining the testing machine with computerized system and relevant software, a reliable control and registration of the test parameters could be achieved.

The proposed method is applied to the characterization of metastable Cr-Ni austenitic sheet alloy that has excellent mechanical and corrosion resistance properties. The material is gaining more interest for its combination of formability and high strength after forming. Till now, there has been no systematic study of the effect of the plastic response of this anisotropic material during biaxial tensile straining on the metal sheet and its mechanical properties. Furthermore, there is a lack of information concerning the correlation between the mechanical response, martensite transformation and microstructural changes during such loading. This problem is a crucial issue for forming process of sheet alloys in automotive industry where complex loading in multiple steps is used to achieve complex shapes. This paper explores the mechanical properties and characterizes the structure of the Cr-Ni austenitic sheet alloy after biaxial tensile straining by hydraulic bulging.

#### 2. MATHERIS AND METHODS

#### 2.1. Sample preparation

Specimens with Ø100 mm diameter were cut from sheet metal alloy X5CrNi18-10 (AISI 304, DIN 1.4301, SUS 304, GOST 07Ch18N10) with chemical composition referred to in [6] and dimensions 1000 x 2000 mm and thickness of  $t_0$ =0,8 mm. In order to retain

the material in place and to prevent the entry of additional material in the examined area, initially a channel was pressed on each sample by testing machine *MC-2000*. The material was tested in as-received conditions.

The general view of test unit for biaxial tensile straining by hydraulic bulging together with a tested sample is shown on *fig. 1. a.* The sheet specimen was fixed to the die with a circular hole (*fig.1.b*) and was plastically deformed by hydraulic bulging. During the test a software product *"HFM"* (Hydro Forming Materials) was gathering the pressure and bulging height values amendment. The test was carried out on equal steps of bulging heights – regular intervals of 5 mm. The test was performed in constant strain rate (0,21 mm/min) and at ambient temperature.



Fig. 1. Test unit for biaxial tensile straining by hydraulic bulging of X5CrNi18-10: a) general view; b) a scheme of the experimental testing unit with a round die [4];

The real stress and strain of the hydraulic bulged anisotropic material are determined according to the equations (1) and (2), respectively:

$$\overline{\sigma}_{anisotr.} = \overline{\sigma}_{isotr.} \sqrt{\frac{2}{1+R}}$$

$$\varphi_{anisotr.} = \frac{\varphi_{isotr.}}{\left[2 - \left(\frac{2R}{R+1}\right)\right]^{1/2}}$$
(2)

where: R - coefficient of average normal anisotropy.

#### 2.2. Sample characterization

The cross section **microstructure** of the sample was carried out using optical microscope *Epytyp-2* and high-resolution digital camera *Zuzi scope*. The specimens were prepared by fine-polishing and etching with concentrated acids HCl and HNO<sub>3</sub> premixed in 3:1 ratio and Kalling's №2 reagent.

**X-ray diffraction (XRD)** measurements were carried out in X-ray diffractometer URD-6, Germany, using Fe-K<sub>a</sub> radiation, operating at 30 kV and 20 mA in the angle range of 40° - 115° (20), 20 step scan of 0,05° with a counting time of 1 s and Nal detector with pulse discrimination. The diffraction patterns for each compound were analyzed by PCPDFWIN JCPDS, v.2002. The grain size was derived from the width of the diffraction peaks according to the equation 3:

$$d = \frac{0,89.\lambda}{\beta.\cos\theta.\cos\frac{\theta}{\lambda}}$$

where d – grain size;  $\lambda$  – FeK<sub>a</sub> wavelenght;  $\theta$  - diffraction peak;  $\beta$  – half-width of the diffraction peaks.

The **hardness** ( $HV_3$ ) of each zone the test specimen had been measured by indentation, using *Wilson Wilpert Hardness tester* with Vickers indenter tip. The indentations were applied for each specimen in the rolling direction (RD) and perpendicular to the RD, under a load of 3 kg.

#### 3. RESULTS AND DISCUSSIONS

The results from the biaxial tensile straining by hydraulic bulging show that the maximum pressure level without loss of resistance and rupture exceeds 22,41 MPa in a bulging height of over 35 mm (fig. 2 a – red curve) [4]. The other diagram from the stepped deformation (fig. 2 a – green curve) is superimposed and compared to first curve of analysis. The results show that both curves practically coincide. The increase of strain causes of plastic-induced change in the magnetic permeability of the sample. The biaxial straining of the sheet material causes degrees of deformation close to 60 % (fig. 2. b).



Fig. 2. Indicator diagrams of hydrostatic pressure – bulging height curves of X5CrNi18-10 sheets (diameter of the die Ø100 mm) indicating the bulging up to destruction (red curve) and stepped deformation (interrupted test) from 10 mm to 35 mm (green curve) b) true strain – true stress curve for the hydraulic bulged material.



Fig. 3. Test sample (general plan view with labelled 4 zones) and its initial microstructure. Each zone is examined in the RD and cross to the RD.

Both curves display serrated flow (type B - irregular with smaller amplitudes that could be attributed to the slow strain rate) during the deformation clearly apparent to the stepped deformed sample at pressure levels over 9 MPa. The scaling behaviour of diagrams could be explained by the microstructural changes in the steel. In order to facilitate the microstructural observations, the hemispherical sample is divided in several zones, shown on *fig.3 a.*, observed at the RD and cross to the RD. **Zone A** 

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Fig. 4. Optical microstructure images of the zones A, B, C and D: a) in the RD; b) cross to the RD. The microstructure consists of aligned bands of bright and gray (with many slip bands) austenitic grains and darker strain induced martensite-containing grains.

The plastic serrated flow of the face-centred-cubic sheet alloy could be attributed to the so-called Portevin-Le Chatelier (PLC) effect and the mechanically induced phase transformation of the metastable austenitic grains. It is known that the collective motion of dislocations within grains and across grain boundaries could cause the observed unsteady flow [7]. The interstitial carbon atoms even at room temperature migrate and form Cottrell atmosphere around dislocations. When the solute atoms have inhomogeneous distribution, they are pined as obstacles by mobile dislocations [8] and the mobile dislocations pile-up and are arrested at localized forest dislocations, then aging or phase transformation occurs [9]. In this Cr-Ni steel, because of the low carbon content, the PLC effect is expected to be less pronounced than the strain phase transformation. The process of aging segregates the dislocations and limits their movements – a phenomenon known as DSA (dynamic strain aging).

The metallographic images (*fig. 4*) in all zones are made mainly along the middle line of the cross-section of the specimen where the signs of the plastic deformation are the greatest. In this area the deformation is more pronounced because of the prior plastic anisotropy of the sheet clearly seen in the rolling direction (RD). As the bulged material is subjected to dominant tension at the top area and dominant compression at the inner surface, those differences will be examined in our next studies.

The structure observed at zone A is very similar to that detected in the uniaxial tensile test of the same sheet material [6]. This microstructure demonstrates a higher volume fraction of austenite matrix for zone A. At higher strains, heading toward the pole (zone B), regular parallel bands form at a characteristic angle with the tensile in the specimen. Gradually approaching the pole, the grains become finer and textured. The externally applied stress and lattice deformation lead to mechanically induced twins and martensitic transformation in the metastable austenitic steel [10]. The presence of  $\alpha'$ martensite explains the magnetic behaviour of the steel. The strain induced transformation is influenced by the orientation, size effect and carbon content in the grains (lower carbon content induces lower stability) [11]. The refining of the structure is obvious but the grain size is hard to be determined because of the extensive dislocation formation, creating subgrains and the martensite formation. In all of the observed zones, the austenite grains are randomly distributed and much more deformed than the martensite. Because the hydrostatic stress in biaxial mode is higher than that of uniaxial deformation mode, the austenite especially near the pole (zone C and D) is elongated and bended while the volume expands after martensite formation. Then the stresses are accommodated in austenite which makes it more unstable and thus more sensitive to phase transformation. However, the cracks or austenitic boundary separation that were found in the same stainless steel under uniaxial tensile test [6], are not found here. Furthermore, it can be considered that the initiation and propagation of cracks are suppressed by the formation of more martensite plates during deformation because the stress concentrations may be relaxed by the formation of preferential martensite variants in stress-concentrated regions [13]. While the uniaxial direction restricts the transformation movements, the biaxial tensile state allows more grains to participate in the strain induced phase transformation.

At low strain near to the flange the darker bands appear at random in the polygonal austenitic grains and on different planes but with straight, nearly parallel orientation (*fig. 5.a and b*). As the deformation slip bands travel only a short distance, their propagation incorporates coupling between non-uniform localized plastic strain, which under certain conditions propagates along the sample and relaxation of the internal stresses with available reloading time occur. When the internal stresses are not totally relaxed, the nucleation of a new band near to the previous one [9], twinning or phase transformation is favoured.

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Fig. 5. Optical microstructure images of slip bands and martensite morpholohy in the different zones: a) non-deformed zone; b) zone A; c) zone B; d) zone C; e) zone D.

The strain-induced matrensite formation starts even at the outermost areas (zone A) of the pole. The martensite plates have acicular morphology and are easily differentiated from one another. At the medium area (zone B) the dark-looking bands exhibit a non-continuous propagation along the tested sample and they are firmer and closely arranged but separated from one another at greater distances (*fig.5 c*). In this zone the strains do not follow a long range planarization, as in the uniaxial tensile tests. As seen, the sites of strain induced martensite nucleation are the intersection points of the slip bands, which was observed from other authors [13, 14]. It would appear that the lack of planarization in the plate microstructure correlates with the alternating tensile strength. Near to the pole the high strain propagates the slip bands continuously and the transformed units are smaller and closely packed but in multiple directions (*fig. 5 d and e*) probably due to the increased amount of simultaneous slips in the grains. It seems that

these bands are able to cross the grain boundaries at higher levels of biaxial stress. Deformed mechanical twins also are seen (*fig.* 5 e) in spite of the crystal rotation. Therefore, the unsteady flow at higher strains (hydrostatic pressure values) could be explained by the discrete stepwise band nucleation and phase transformation.

The XDR pattern (fig. 6) of the as-received sample shows peaks of fcc-austenitic phases. In the deformed zones A, B, C and D a mixed austenite-martensite phase composition is present. Going to the pole, a clear trend could be observed, in particular: the diffraction lines of all plastically deformed zones (A, B, C and D) are obviously broadened and simultaneously show decreased intensity. This fact is another evidence for the increased strains and the refinement of the grains. As the deformation extend proceeds, the plastic strain accumulated in the austenitic and martensitic phases increases. The only martensite found is bcc  $\alpha$  -phase that is known to nucleate at dislocation pileups [15], mechanical twins or shear bands. Therefore, the biaxial tensile straining by hydraulic bulging is more beneficial for  $\alpha$  -martensite formation than uniaxial tension. Higher martensite fraction may be attributed to enhanced transformation mechanisms because of the biaxial stress performance and greater volume expansion. According to the XRD analysis there is a lack of hexagonal *ε*-martensite, which presence is considered as a result of not enough driving force energy that leads to the conclusion that the sequence of events in the austenitic material during hydrauling bulging straining is as follow: fcc-austenite  $\rightarrow$  twinnning/shear bands  $\rightarrow \alpha$ -martensite. The presence or absence of an intermediate *ε*-martensite (hcp-phase) should be verified by other more sensitive methods.





As estimated from the line broadening of the  $\gamma$ -Fe,Ni,Cr <sub>(220)</sub> (*fig.* 6), the average grain size of top layer of the as-received sheet alloy is about 23,9 µm, while it is 23,5 µm, 22,01 µm, 19,7 µm and 18,05 µm of zones A, B, C and D of the hydraulic bulged sample, respectively. In addition, the initial coarse grained structure is more susceptible to deformation twinning [16] and martensitic transformation [17] because the austenite stability decreases with increasing grain size. It should be mentioned also that the surface can suffer from stress relaxation or different stress (compression or tension) and there would be differences in texture and size between surface and bulk sheet material (neutral

line). The small test rate does not permit the sample to be heated up which increases the driving force for the matrensite formation. The resulting diffraction peaks are not so intense so that a reliable analysis of the volume fractions of both phases could not be done with a good precision.



Fig. 7. Cross sectional hardness values of the tested steel in the RD and perpendicular to the RD.

The hardness values are measured in the neutral line in the cross section of the cuts of the samples. Due to the high plastic strain, smaller grain size and increase of the fraction of hard martensite, the hardness values of the pole are the highest (*fig. 7*). Moving away from the pole the values of the hardness smoothly decrease that proves the ability of the material for further deformation (not exhausted ductility) in this area. It could be observed that the deformation strain in the rolling direction near to the pole and near to the hemisphere basis is greater than the strain in perpendicular direction for the hydraulic bulged austenitic sheet alloy. In the middle areas the hardness values in both directions. The nature of the amendment of the hardness values does not show any substantial differences. The presence of more martensite contributes to the overall straining of the mixed austenite-martensite structure. The retained plasticity and ductility of the sheet material demonstrates that the PLC effect that drastically decreases these properties, is likely to have less impact on the strengthening mechanism of this material.

#### 4. CONCLUSIONS

It has been found that the hydraulic bulging by straining at room temperatures and slow rates results in deformation induced phase transformation. The deformation microstructure of the austenitic steel reveals that long planar matrensite plates are formed in the outermost regions of the pole, while short-range planarity plates with different orientation are typical for the near pole zones. The sizes of these structures in terms of thickness and phase spacing decrease from the outermost areas towards the pole in both directions. The martensite transformation does not occur homogenously in the different zones of the bulged material. The amount of martensite transformed is increased with increases in hydraulic strain and stress levels. The fine-sized martensite plates formed during biaxial hydraulic straining is expected to be resistant to cracking. The strengthening of the hydraulic bulged zones originates mainly from the grain refinement effect and the mechanically induces phase transformation. The martensite transformation is the prevailing process in the biaxial tensile straining by hydrauling bulging which enchances the strength of the metastable austenitic sheet alloy.

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#### The paper is reviewed.

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## **DIPLOMA**

The Programme Committee of the Scientific Conference RU&SU'15 AWARDS THE CRYSTAL PRIZE "THE BEST PAPER" to Emil Yankov, Maria Nikolova, Vanya Zaharieva authors of the paper

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