ASYMMETRIC CRYOROLLING OF THE AUSTENITIC STAINLESS STEEL AISI 304

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Abstract

The ultra-fine grained austenitic AISI 304 stainless steel sheets were prepared using various rolling techniques. The room temperature symmetric rolling (SR) as well as the sub-zero temperature cryorolling (CR) were performed. In addition to conventional rolling techniques the asymmetric rolling (ASR) and the asymmetric cryorolling (ACR) experiments were set up. Different rolling techniques were compared with regard to the resulting mechanical properties and feasibility. In comparison with the symmetric rolling, the asymmetric rolling process imposes additional shear strain to the normal plane strain. A large plastic strain has a direct influence on the process of the grain refinement. Cooling the steel to the sub-zero temperatures supresses the dynamic recovery and recrystallization, thus improving the grain refinement process. A high velocity ratio ACR (1:2.3) and the CR (liquid N_2) techniques give rise to the austenitic stainless steel with high values of the yield strength, the tensile strength and the ductility at the same time.

Keywords: Cryorolling, asymmetric rolling, austenitic stainless steel, mechanical properties, grain refinement

1. INTRODUCTION

In recent years a significant effort has been made to develop a bulk ultrafine-grained (UFG) metals using the phenomenon of severe plastic deformation (SPD). UFG metals usually possess unique and favourable properties, e.g. higher tensile strength and ductility. A number of studies have reviewed the application of SPD [1-5]. These studies were focused mainly to a fabrication of non-ferrous metals in rather small, even laboratory scale [6]. The laboratory scale methods attempted to obtain UFG structures via equal channel angular pressing, accumulative roll bonding or high pressure torsion techniques. However, the majority of all metal products is produced using various rolling processes. The present study is focused on manufacturing UFG austenitic stainless steel AISI 304 using more profound rolling techniques [6]. Austenitic stainless steel AISI 304 is commercially available and frequently used construction material due to its good corrosion resistance and ductility. However, both the yield strength and the ultimate tensile strength is rather poor (approx. 200-300 MPa, 500-600 MPa respectively), mainly thanks to the soft face centred cubic (fcc) γ-phase. The strength of the austenitic stainless steel can be improved by the grain refinement, solid solution strengthening and work-hardening [7,8]. The grain refinement has been achieved by adopting thermo-mechanically (TMCP) controlled processing. However, recent studies [9,10] have indicated that the limitation in grain refinement achievable by TMCP can be overcome by severe plastic deformation, and thereby UFG structures can be acquired. The CR, ASR, ACR techniques are considered as SPD processes [11,12]. The presented experiment compares above mentioned rolling techniques with conventional SR with regard to resulting mechanical properties and feasibility. Asymmetric rolling techniques utilize a different velocity of working rolls. The velocity ratio ranging from 1.1 up to 4 has been used in most of the recent studies [6,11]. Both the hot and the cold ASR techniques have been applied to various ferrous and non-ferrous materials [13,14]. Due to the different circumferential speed of working rolls friction forces act in the opposite direction at the upper and lower work roll surfaces, thus generating additional shear strains. This additional shear strain may cause structural changes of the rolled material which can be beneficial for material properties. During the rolling process the dynamic recovery may occur due to the increased deformation resistance at higher strain. Decreasing the deformation temperature leads to suppression of dynamic recover that results in higher defects density and consequently higher strength. AISI 304 steel has a fully austenitic structure. However, the application of plastic deformation or cryogenic treatment can lead to formation of martensite in metastable austenitic steels [6]. In general, face centred cubic metals exhibit higher work-hardening rates. Both the stable dislocation interactions and very fine deformation twins contribute to the high flow stress.

2. EXPERIMENTAL

A commercially available AISI 304 austenitic stainless steel (18/8) sheet of the 6 mm thickness was used. The sheet was cut into 100 mm wide strips. SR and CR experiments were performed on the COMTES FHT rolling mill using the four-high rolling mill setup. Diameters of the working rolls and the back-up rolls respectively are 240 mm and 650 mm respectively (**Figure 1**). The multi-pass unidirectional SR and CR was carried out. The input AISI 304 sheet in 6 mm thickness was rolled by approximately 5 % reduction at each pass down to 4 mm (i.e. 30 % accumulated reduction), 3 mm (i.e. 50 % accumulated reduction) and 2,4 mm (i.e. 60 % reduction) respectively. The ASR and ACR experiments were carried out incorporating the four-high rolling mill rolls and the two-high rolling mill roll with the diameter of 550 mm. The CR and ACR samples were cryorolled after immersing them in liquid nitrogen before each pass. Asymmetrically rolled samples were flipped horizontally before each pass due to the curvature of the as-rolled sheets. The ASR and ACR experiments did not run smoothly. Due to friction stresses generating additional lateral forces and insufficient support from the back-up roll, the wiggling effect occurred (**Figure 1**). Instead of continuous and smooth flow through the rolling gap the material skipped in an abrupt manner. Due to this fact deformation was not uniform across the cross-section. However, the surface layers were severely deformed. The attempt to use a lubricant to prevent wiggling behaviour failed. The cryorolled samples, i.e. CR and ACR, exhibited almost twice as high deformation resistance in comparison to symmetrically rolled samples, i.e. SR and ACR. **Figure 4** illustrates different deformation behaviour of the room temperature and cryorolled samples. Due to the poor control of the ASR and ACR experiments the respective rolling passes can't be compared to those of SR and CR experiments. Thus, unfortunately, the lower rolling forces and energy savings, being the advantages of asymmetrical rolling, couldn't be confirmed.

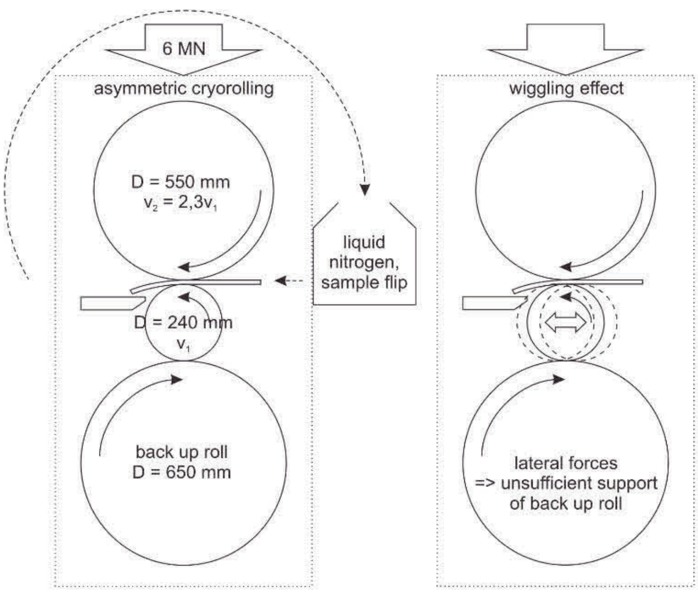

Figure 1 Left: Asymmetric cryorolling (ACR). Right: Wiggling effect due to the insufficient support from the back up roll.

3. RESULTS

The microstructure was revealed using the Glyceregia etchant and light microscope NIKON ECLIPSE MA200 equipped with differential interference (DIC) enabling high contrast image observation. The cryorolled material shows more deformed grain structure as opposed to the room temperature rolling. The grains are elongated along the direction of rolling. The microstructure shows slip bands, deformation twins and the presence of δ-ferrite as well (**Figure 2**). The attempt to explore both the structure and the texture evolution using Electron backscatter diffraction (EBSD) technique failed due to the extremely deformed structure and insufficient resolution of EBSD camera. The scanning electron microscopy (SEM) revealed extremely deformed surface layer. The structure is presumably austenitic. The expected martensite transformation has not been confirmed. The austenitic matrix seems to be stable at all deformation stages used in the experiment.

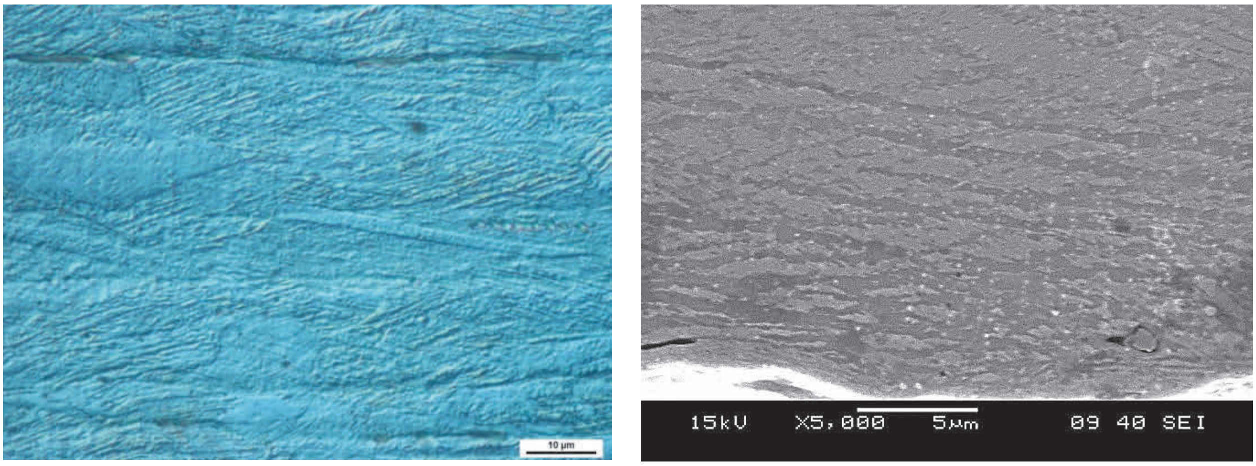

Figure 2 Left: DIC image of the C4 sample - inner layer. Right: SEM image of the AS4 sample - surface.

Tensile tests were done according to the standard CSN EN ISO 6892-1. Three samples were tested at each deformation step. Prior to testing, specimen dimensions were measured and an original gauge length for the elongation determination was marked on each specimen. This gauge length was set to meet requirements of the standard for the elongation A_5. After each test, a proof stress $R_{p0,2}$ was determined as well as ultimate tensile stress R_m. The final gauge length was also measured after each test and elongation after fracture A_5 was evaluated as well as the cross section reduction Z. Tests were done on electro-mechanic testing machine Mays equipped with hydraulic grips. Strain was measured by means of DIC system (Digital Image Correlation) Mercury in 2D configuration. Geometry of the specimens is depicted in **Figure 3.** All specimens were produced by water jet cutting perpendicularly to the rolling direction.

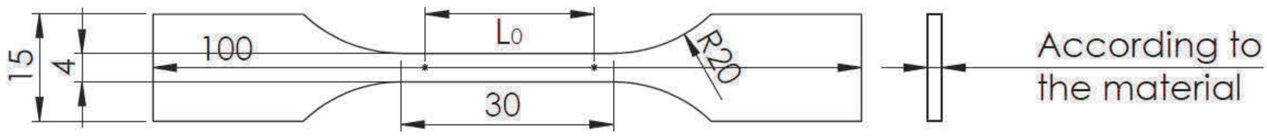

Figure 3 Tensile test specimen geometry. Extensometer length L_0 varies with the thickness of the specimen.

Table 1 summarizes the results of the mechanical testing of the original austenitic stainless steel and SR, CR, ASR, ACR rolled material. The hardness enhancement follows from diminishing grain size and from the accumulation of large plastic strain achieved by cryorolling. As mentioned before, a cryogenic temperature helps to suppress dynamic recovery and recrystallization, leading to high dislocation density. As the strain increases deformation twins are created in the austenite structure which is beneficial for the grain refinement. HV30 hardness of the asymmetrically rolled material is 20 HV higher in comparison to symmetrically rolled material mainly due to accumulating of larger plastic strain in surface layers. **Figure 4** shows the engineering stress-strain curves from tensile tests of the austenitic stainless steel before and after the SR, CR, ASR and ACR rolling. Annealing was performed at 750 °C with the hold time 5 minutes and cooling in the water. The input material shows low proof stress of 269 MPa and the tensile strength of 626 MPa. The proof strength and the tensile strength increases rapidly in the early stages. As the accumulated deformation reaches certain level the strength increases slightly and become stable (**Figure 5**). The ratio of proof strength to tensile strength increases from 0.43 @ 0% reduction up to 0.91 @ 60% reduction and become stable as well (**Figure 6**). This fact indicate that the austenitic matrix is stable and no deformation-induced martensitic transformation occurred. The grain refinement effect generated at cryogenic temperatures can be documented on increased ductility while the proof stress and tensile strength remain or even increase (**Table 1**). Moreover, the significant mechanical enhancement are achieved after much lower accumulated reduction when using cryogenic temperatures.

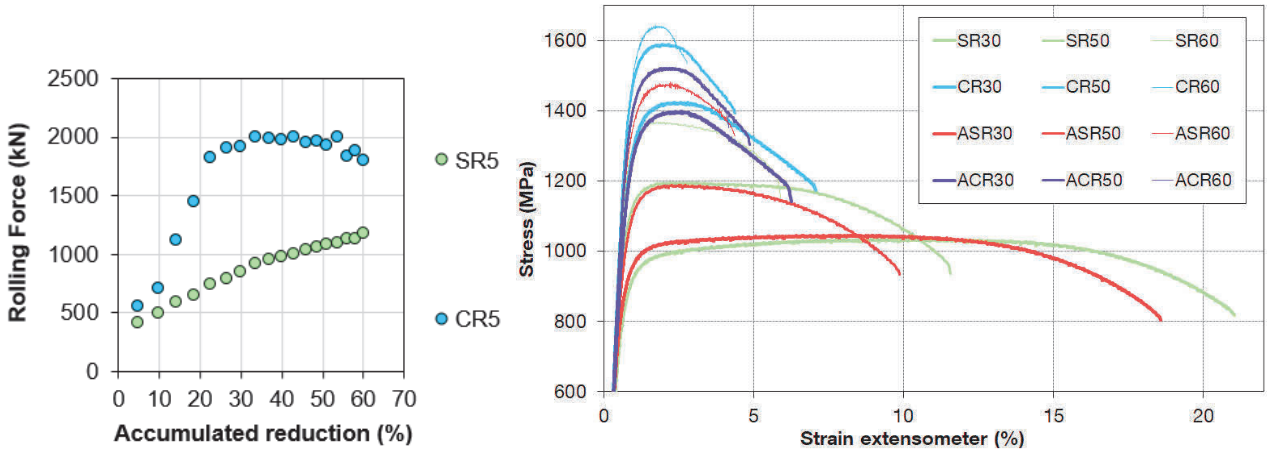

Figure 4 Left: Rolling force during 5 % reduction steps. Right: Engineering stress-strain curves.

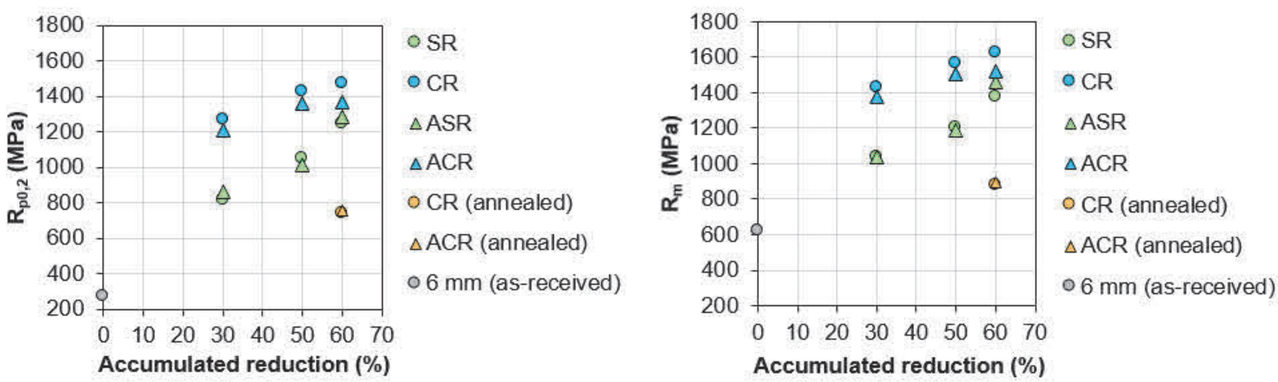

Figure 5 Left: Proof stress. Right: Tensile strength.

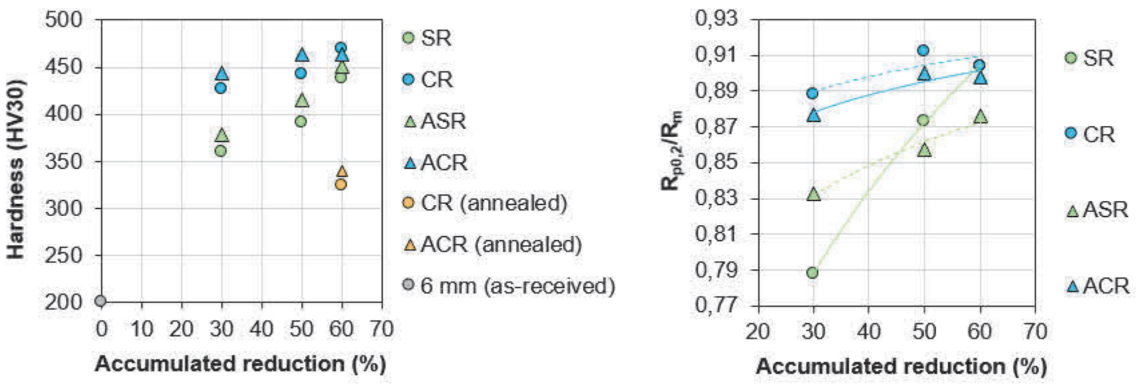

Figure 6 Left: Hardness HV30. Right: Proof stress to tensile strength ratio.

4. DISCUSSION

The combination of cryogenic temperatures and large plastic deformation directly effects the process of grain refinement thus significantly enhancing mechanical properties. The tensile tests results of the respective symmetric rolling experiments and asymmetric rolling experiments are comparable to each other. Due to the high velocity ratio and friction-induced lateral forces together with insufficient stiffness of the working roll, the wiggling effect occurred. The wiggling of working roll prevented the shear to act uniformly across sheet cross-section. Thus, only surface layers (approx. 100 μm) were influenced by additional shear strain. Locally accumulated strain manifests itself via increased surface hardness. To be able to fully exploit the potential of asymmetric rolling, much stiffer working roll has to be incorporated. Moreover, the velocity ratio seems to be inefficient and high. The attempt to use a lubricant had no direct effect. The austenitic matrix of AISI 304 stainless steel seems to be stable at least up to the 60% accumulated reduction. No deformation-induced transformation was confirmed. The engineering stress-strain curves indicate no significant increase in strength due to the deformation-induced martensite transformation. The strengthening is a result of the work-hardening and the grain refinement. However, the structure is unclear, especially at large deformations. Mainly because of extremely deformed structure, poor etching ability of the soft austenite and insufficient resolution of used EBSD camera. The XRD analysis should be carried out instead.

Table 1 Average values of tensile tests measurements

Specimen	HV30	$R_{p0,2}$ (Mpa)	R_m (MPa)	R_p/R_m	Z (%)	A_5 (%)
As-received	200	269	626	0,43	74,6	73,3
SR30	360	819	1039	0,79	49,0	19,2
SR50	391	1053	1206	0,87	40,9	12,8
SR60	438	1248	1381	0,90	25,1	9,6
CR30	427	1270	1431	0,89	23,9	8,0
CR50	442	1429	1566	0,91	16,0	4,2
CR60	470	1471	1628	0,90	9,1	5,5
ASR30	378	864	1037	0,83	43,9	17,6
ASR50	415	1015	1185	0,86	45,3	13,1
ASR60	451	1282	1464	0,88	16,2	8,6
ACR30	444	1210	1380	0,88	31,4	8,5
ACR50	463	1359	1509	0,90	14,6	5,5
ACR60	463	1364	1519	0,90	16,4	4,9
SR60 HT	322	785	951	0,83	50,4	38,1
CR60 HT	324	744	879	0,85	55,6	38,0
ACR60 HT	340	758	894	0,85	54,6	35,0

5. CONCLUSIONS

The room temperature SR, CR and cryogenic temperature ASR and ACR rolling experiments were set up and carried out to examine the plastic deformation behaviour of austenitic stainless steel AISI 304. The asymmetric rolling experiment utilized the high velocity ratio of 2.3. The results of respective symmetric and asymmetric experiments are almost the same thanks to the wiggling effect of the working roll. The dynamic recovery and recrystallization was suppressed using the cryogenic temperatures. Thus, significant grain refinement was achieved enhancing the mechanical properties greatly. Highly elongated austenitic grains show slip bands and deformation twins. No deformation-induced martensitic transformation was confirmed.

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REFERENCES

[1] PALÁN, Jan, PROCHÁZKA, Radek, ZEMKO, Michal. The microstructure and mechanical properties evaluation of UFG Titanium Grade 4 in relation to the technological aspects of the CONFORM SPD process. *Procedia Engineering*. 2017. vol. 207, pp. 1439-1444.

[2] PALÁN, Jan, ŠUTTA, Pavol, KUBINA, Tomáš, DOMÁNKOVÁ, M. Effect of severe plastic and heavy cold deformation on the structural and mechanical properties of commercially pure titanium. *Materiali in Tehnologie*. 2017. vol. 51, iss. 5, pp. 849-853.

[3] DUCHEK, M., KUBINA, T., HODEK, J. and DLOUHÝ, J. Development of the Production of Ultrafine-grained Titanium with the Conform Equipment. *Materiali in Tehnologije*. 2013. vol. 47, pp. 515-518.

[4] KARLÍK, M., SLÁMOVÁ, M., HOMOLA, P., SLÁMA, P., CIESLAR, M. Accumulative Roll-Bonding (ARB) of Sheets of Aluminium and its Commercial Alloys AA8006 and AA5754 at Ambient and Elevated Temperatures. *Materials Science Forum*. 2007, vols. 546-549, pp. 767-774.

[5] HOMOLA, P., SLÁMOVÁ, M., SLÁMA, P., CIESLAR, M., KARLÍK, M. Preparation of ultrafine-grained twin-roll cast AlMg3 sheets by accumulative roll bonding. *International Journal of Materials Research*. 2009. vol. 100, no. 6, pp. 863-866.

[6] YU, H. Liang, LU, C., TIEU, A. Kiet, LI, H. Jun., GODBOLE, A. and ZHANG, S. Hong. Special rolling techniques for improvement of mechanical properties of ultrafine-grained metal sheets: a review. *Advanced Engineering Materials*. 2016. vol. 18, iss. 5, pp. 754-769.

[7] BHADESHIA, Harshad K. D. H. and HONEYCOMBE, Robert. Steels: Microstructure and Properties. 3rd edition. Elsevier, 2006, chapter 12, pp. 259-286.

[8] BARNA, Roy, RAJESH, Kumar, JAYANTA, Das. Effect of cryorolling on the microstructure and tensile properties of bulk nano-austenitic stainless steel. *Materials Science & Engineering A*. 2015. vol. 631, pp. 241-247.

[9] MISRA, R. D. K., NAYAK, S., VENKATASURYA P. K. C., RAMUNI, V., SOMANI, M. C. and KARJALAINEN, L. P. Nanograined/Ultrafine-Grained Structure and Tensile Deformation Behavior of Shear Phase Reversion-Induced 301 Austenitic Stainless Steel. *Metallurgical and Materials Transactions A*. 2010. vol. 41A, pp. 2162-2174.

[10] XIONG, Yi, HE, Tiantian, WANG, Junbei, LU, Yan, CHEN, Lufei, REN, Fengzhang, LIU, Yuliang, VOLINSKY, Alex A.Cryorolling effect on microstructure and mechanical properties of Fe-25Cr-20Ni austenitic stainless steel. *Materials and Design*. 2015. vol. 88, pp. 398-405.

[11] FAJFAR, Peter, LAH, Alenka Š., KRANER, Jakob and KUGLER, Goran. Asymmetric rolling process. *Materials and Geoenvironment*. 2017. vol. 64, iss. 3, pp. 151-160.

[12] LU, C., LI, J. & FANG, Z. Effects of asymmetric rolling process on ridging resistance of ultra-purified 17%Cr ferritic stainless steel. *International Journal of Minerals, Metallurgy, and Materials*. 2018. vol. 25, iss. 2, pp. 216-225.

[13] ROMBERG, J., FREUDENBERGER, J., WATANABE, H., SCHARNWEBER, J., ESCHKE, A., KÜHN, U., KLAUß, H., OERTEL, C.-G., SKROTZKI, W., ECKERT, J., SCHULTZ, L. Ti/Al Multi-Layered Sheets: Differential Speed Rolling (Part B). Metals. 2016. vol. 6, iss. 2, no. 31.

[14] HAMAD, Kotiba, KO, Young G. Effect of roll speed ration on microstructure evolution and mechanical properties of 0.18wt% carbon steel deformed by differential speed rolling. *Material Letters*. 2015. vol. 160, pp. 213-217.