

Correlation between Heat Treatment, Microstructure and Properties of Trip-Assisted Steels

A. Talapatra, N. R. Bandhyopadhyay, J. Datta

Abstract—In the present study, two TRIP-assisted steels were designated as A (having no Cr and Cu content) and B (having higher Ni, Cr and Cu content) heat treated under different conditions, and the correlation between its heat treatment, microstructure and properties were investigated. Micro structural examination was carried out by optical microscope and scanning electron microscope after electrolytic etching. Non-destructive electrochemical and ultrasonic testing on two TRIP-assisted steels was used to find out corrosion and mechanical properties of different alter microstructure phase's steels. Furthermore, micro structural studies accompanied by the evaluation of mechanical properties revealed that steels having martensite phases with higher corrosive and hardness value were less sound velocity and also steel's microstructure having finer grains that was more grain boundary was less corrosion resistance. Steel containing more Cu, Ni and Cr was less corrosive compared to other steels having same processing or microstructure.

Keywords—TRIP-assisted steels, heat treatment, corrosion, electrochemical techniques, micro-structural characterization, non-destructive (ultrasonic) technique.

I. INTRODUCTION

THE automotive industries pay a great deal of attention to development of the new classes of steels with enhanced combination of strength–formability for more affordable, safe and fuel-efficient vehicles. From the history of steel development for vehicle, we can see that there were many kinds' steels, which had been developed and used. From the 1950's to the 1960's, the rimmed steel was researched and used. From the 1960's to 1970's, many killed steels were developed and employed. From the 1980's to the 1990's, to highly improve the formability of sheet steel, the research of auto steel was mainly centralized in interstitial-free (IF) steel. By the 21st century, most projects of auto steel focused on developing new kinds of HSS and new style steels to meet the needs of the plan of ultra light steel auto body (ULSAB). These steels include double-phase (DP) steel, TRIP-steel and TWIP-steel [1]. Transformation induced plasticity (TRIP) aided steels and twinning induced plasticity (TWIP) aided steels are the promising candidates fulfilling such demands [2]. The main difference between TRIP-assisted and TWIP steel is that the austenite in the former is stable on cooling but

not under mechanical load, i.e. phase transformation happens when the material is loaded. In contrast, there is no phase transformation in TWIP steel during cooling or deformation, but the orientation of part of austenite will change due to mechanical twinning. The different behavior of the austenite is attributed to its stacking fault energy (SFE). If SFE is very low ($<20 \text{ mJ/m}^2$), martensitic-induced plasticity is favored [4]. Higher SFE of the order 25 mJ/m^2 suppresses martensitic phase transformation and favors mechanical twinning until SFE values $<60 \text{ mJ/m}^2$ [5], [6]. Steels having a fully austenitic microstructure are called TRIP steels [3]. These steels tend to be rich in nickel and other expensive austenite stabilizing elements. By contrast, austenite is only a minor phase in the overall microstructures of TRIP-assisted steels [7]. Challande and Padilla [8], [9] have investigated the general corrosion resistance of Cr-Ni-C and Cr-Ni-Mo-C TRIP steels, utilizing an anodic polarization testing procedure. They found that 13Cr-8Ni-3Mo-0.25C and 13Cr-7Ni-4Mo-0.25C TRIP steels have both very good mechanical and very good corrosion-resistant properties. There was no information, however, on the resistance of TRIP steels to localized attack (pitting corrosion), especially in chloride solutions, including seawater. Nor was there information on the corrosion resistance of TRIP steels that have been cold-worked and thus partly transformed to Martensite [13], [14]. Keeping in view above study on corrosion behaviors to moderately low carbon steels having Si, Mn, Ni, V, Nb, Mo and/or Cu and Cr, designed and develop for automotive application has been attempted in this study. It is essential to do more study in corrosion behavior and performance of the material in order to evaluate and improve the design, cost effectiveness and reliability of each material used. Khoshnaw [10] studied fatigue strength of low alloy steels in chloride solution. Turnbull [12] studied stress corrosion cracking of stainless steel in chloride solutions. Chen [11] studied mechanical properties of low-alloy steels in atmosphere containing chloride in tension test.

II. EXPERIMENTAL PROCEDURE

A. Materials

Two moderately low Carbon high strength low alloy steels is designated as A and B having composition given in Table I were supplied by DMRL, Hyderabad.

Animesh Talapatra is an assistant professor, MCKV Institute of Engineering (under WBUT), West Bengal, India-711204 (phone: 91-33-26549315/17; fax: 91-33-26549318; e-mail: animesh_talapatra@yahoo.co.in).

N. R. Bandhyopadhyay is a professor, BESU, Shibpur, West Bengal, India. He is now with the school of material science and engineering, BESU, Shibpur, India, (e-mail: nrb@matsc.becs.ac.in).

Jayati Datta is a professor with the Chemistry Department, BESU, Shibpur, West Bengal, India (e-mail: jayati_datta@rediffmail.com).

TABLE I
CHEMICAL COMPOSITION OF MATERIALS

Steel	C	Mn	Si	P	Ni	Cr	Cu	V	Nb	Mo
A	0.11	1.3	0.3	0.02	0.75	0	0	0.03	0.03	0
B	0.10	0.47	0.28	0.02	2.02	0.4	0.6	0.03	0	0.44

B. Heat Treatment

Steels were heat treated as per schedules as shown in below in Table II to develop different microstructure.

TABLE II
HEAT TREATMENT OF STEELS

Serial No	Steels	Heat treatment
1	A1	As received (oil quenched and tempered)
2	A2	Held at 9500C for 1/2hr,air cooled, Held at 7000C for 2 hrs, quenched to 4000C(salt bath), held for 900s and oil quenched.
3	A3	Held at 9500C for half an hour, oil quenched in an oil of 0.123 centistokes viscosity.
4	A4	Held at 9500 C for half an hour then cooled in water
5	A5	Held at 9500C for half hour then cooled in air
6	A6	Held at 9500C for 1/2hr., and then cooled in furnace by switching off.
1	B1	As received (water quenched and tempered)
2	B2	Held at 9700C for 1/2hr,air cooled, Held at 8000C for 2 hrs, quenched to 4000C (salt bath), held for 900s and oil quenched.
3	B3	Held at 9700 C for half an hour , oil quenched in 0.123 centistokes viscosity
4	B4	Held at 9700 C for half an hour, cooled in water
5	B5	Held at 9700 C for half an hour , then cooled in air
6	B6	Held at 9700C for 1/2hr., and then cooled in furnace by switching off.

C. Micro- Structural Characterization

The micro structural characterizations of the steels were carried out using an OLYMPUS CK40M-CP optical microscope. The optical metallographies of all these samples were carried out in the usual way. Samples were cut from plates of these steels. These samples were polished on polishing wheel after 1 to 6 (rough to fine) emery papers. The polished surface appeared like mirror having no scratches and the etchant was used 2% nital. The washed and dried samples were observed carefully in Microscope at different magnification and some selected photomicrographs were taken. TRIP-aided steel are composed of four phases forming a very fine microstructure which makes their observation difficult. For light microscopy observation, some specific etchants have been tested. The phase analyses ASTM E 566 1245 have been done by using Olysia m3 software. Samples were prepared as like as preparation of sample for optical microscope observation. Then images were taken in optical microscope. The acquired images of a multiphase object were taken for analysis. Phase analysis will be conducted on a gray-value image. The image is selected and threshold is set to define the gray value ranges for the separate phases. The OLYSIA software created a measurement sheet showing the absolute area and proportional area (in %) of all the phases. The measured values are taken.

D. Electrochemical Study

Samples were taken in an area of 0.204 square cm size coupons for performing potentiodynamic polarization studies in de-aerated condition in cells with three electrode configuration and using aqueous saturated calomel SCE (W) as the reference electrode and Pt foil as counter electrode. Linear sweep voltammetry was performed with the help of AUTOLAB 12 PGSTAT, Eco Chemic B.V (the Netherlands) at 0.5 mV/s scan rate within the potential range of -1500 mV to the cathodic potential of 650 mV vs.SCE. Potential scans were conducted in de-aerated conditions by purging the solution with nitrogen for 10min. Tafel analysis was performed to determine the corrosion parameters. Electro-chemical Impedance Spectroscopy at the respective OCP value were recorded with the help of AUTO-LAB 12 PG STAT, Eco Chemie B.V (the Netherlands) combined with frequency response analyzer (FRA) module. The sinusoidal perturbation of 5mV amplitude was applied at the cell over the frequency range of 100 KHz to 10MHz. EIS measurements were conducted at open circuit conditions after a steady state potential was attained in aerated solution of different electrolytes at neutral pH. The experiment was performed in three electrode one compartment cell containing the test coupons as working electrode, a large area Pt foil as counter electrode and a saturated calomel reference.

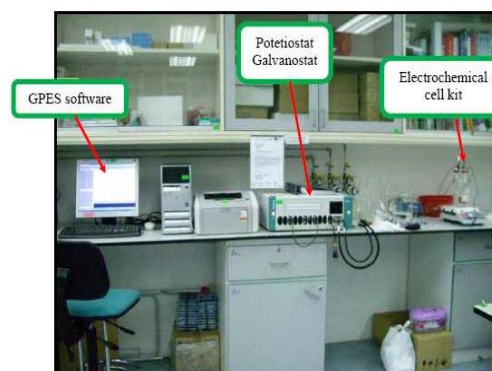


Fig. 1 Electrochemical lab set up

E. Nondestructive Testing Study

The measurement of the ultrasonic velocities was accomplished using a longitudinal and transverse wave transducer. The technique used was the pulse echo based on the direct contact with the sample. To measure the velocities of longitudinal waves, it was used a normal incidence transducer of 5 and 10MHz. The EPOCH4 ultrasonic flaw detector with its high voltage pulse, the quality of its square pulse and selectable narrowband filters is the instrument of choice for this technique. With the values of materials' densities and ultrasonic velocities of longitudinal and transverse waves, the elastic modulus was calculated based on the ASTM E 494-1995 norm (Measuring Ultrasonic Velocity in Materials) using the equation. After the measurements of the longitudinal and transversal wave ultrasonic velocities,

was calculated the elastic modulus of each sample, considering the ASTM E 494-95 norm. According to the ASTM E-494-05 norm, the results for the elastic modulus obtained by ultrasound testing have an error around 1% in comparison to the values obtained by mechanical testing [21], [22]. Elastic modulus is related to the inter-atomic forces and hence indicates maximum attainable strength [15], [16]. There exists a direct mathematical relationship between elastic modulus and ultrasonic longitudinal and shear velocity. These relationships are as follows:

$$\text{Young's Modulus } E = \rho \cdot V_T^2 \cdot (3V_L^2 - 4V_T^2) / (V_L^2 - V_T^2),$$

$$\text{Shear Modulus } G = \rho \cdot V_T^2,$$

$$\text{Bulk Modulus } B = \rho \cdot (V_L^2 - 4 \cdot (V_T^2) / 3),$$

$$\text{Poisson's Ratio } \nu = (V_L^2 - 2 \cdot V_T^2) / (2 \cdot V_L^2 - 2 \cdot V_T^2),$$

$$\text{Fracture toughness } K_{IC} \text{ is given by: } K_{IC} = \sqrt{E' \cdot G_c}.$$

E' is the Young's Modulus; G_c is the strain energy release factor. Kim and Johnson et al. [17] using resonant ultrasonic spectroscopy measured the elastic modulus of ferrite, ferrite pearlite, and martensite microstructures, being the obtained elastic modulus of 211.9, 210.3 and 203.5 GPa and the density values of 7.851, 7.835 and 7.709 g/cm³, respectively. According to Hakan and Orkun [18], in the case of the specimens consisting of pearlite-ferrite, the main difference in the microstructure is the spacing of the cementite lamellae-ferrite, and content and size of ferritic phase. Yield strength is defined as a lower yield point or as the strength at 0.2% offset strain in case of the absence of a yield point [23]. The work-hardening behavior is described using the change in the instantaneous work-hardening exponent n is defined as the following equation evaluated from the true stress-strain curve [19], [20].

III. RESULT AND DISCUSSION

A. Microstructural Characteristic

The microstructure characterization was carried out using optical microscope OLYMPUS CK40M-CP JAPAN. Microstructures of the corroded samples were studied by optical microscope after electrolytic. The morphology of retained austenite has been classified into five different categories: blocky shape enclosed by polygonal ferrite, blocky or layer shape enclosed by acicular ferrite, blocky or layer shape enclosed by bainite packets, interlayer film enclosed by martensite laths. The optical microstructure of as received condition (A1) reveals mostly polygonal ferrite with certain amount of tempered bainite as shown in Fig. 2. The optical microstructure of as received condition (B1) reveals mostly tempered bainitic structure with certain amount of blocky polygonal ferrite as shown in Fig. 4.

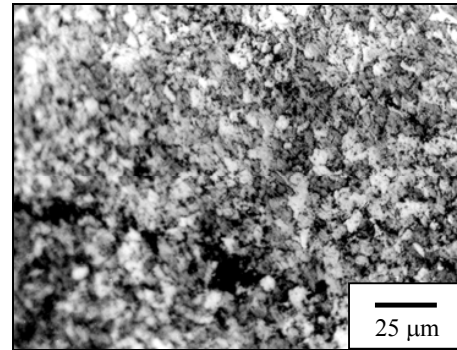


Fig. 2 Optical microstructure of Steel (A1)

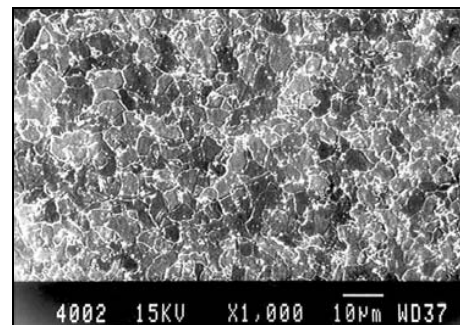


Fig. 3 SEM of Steel (A1)

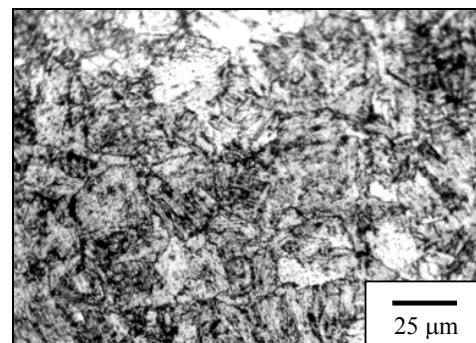


Fig. 4 Optical microstructure of Steel (B1)

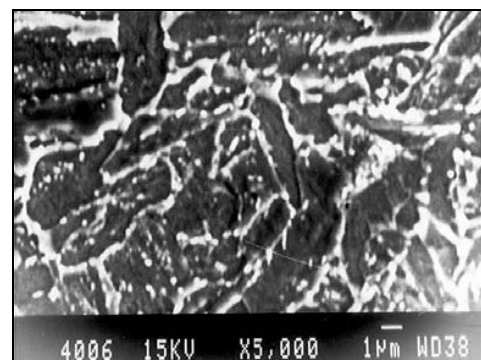


Fig. 5 SEM of Steel (B1)

Post corrosion microstructures, taken for a few samples for both steels, reveal adequate corrosion both in the form of

grain boundary attack and pitting. However steel having high percentage of Cu and Cr, in case of steel B, this corrosion attack is comparatively less in all heat treatment schedules seemingly due to formation of protective oxide layer. All the above microstructure when subjected to image analyzing system, it is seen that high % of martensite and/ or pearlite in ferrite matrix during water and air cooling.

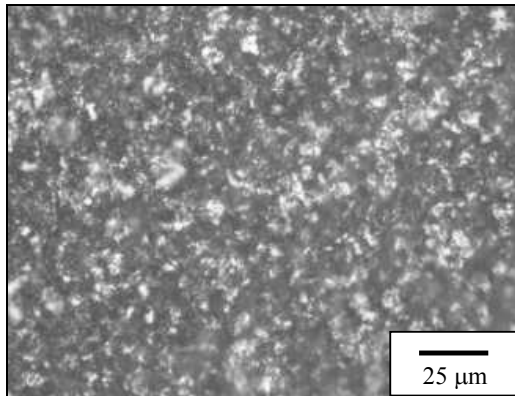


Fig. 6 Optical microstructure of Steel (A1 in 0.1% NaCl)

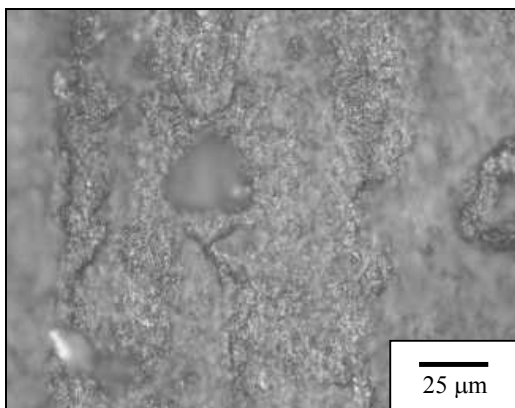


Fig. 7 Optical microstructure of Steel (B1 in 0.1% NaCl)

B. Corrosion Characteristic

Typical polarization plots as shown in (Fig. 8) of A2 and B2 in 0.1% NaCl shows that B2 is characterized with a narrow but distinct passive region while in A2 there is no such region. EIS measurement reveals the Nyquist plot as shown in (Fig. 10) where the diameter of half-circles represents the circuit resistance of the material. While in pure water (0% NaCl) Nyquist plots as shown in are characterized with two subsequent but overlapping semicircles, in Cl⁻ ions containing solution only single half-circles are obtained. In neutral pH when EIS represents dual character A samples are more resistant than B samples $R_p(A) > R_p(B)$ from Tables III & IV. It may be predicted that matrix / grain boundary of A is more reactive than that of B in neutral conditions and that may be due to formation of thicker and stern passive layer (oxide film) in alloy B. This gets disrupted immediately in contact with Cl⁻. In most of A and B samples 10-fold decrease is

observed when exposed to 0.1% NaCl.

TABLE III
CORROSION PROPERTIES (CORROSION RATE (C.R))

Steels	NaCl 0%	NaCl 0.1%	NaCl 1%	NaCl 3%	C.R (mm/yr)
A1	0.36	0.54	5.47	-	
A2	0.12	0.28	2.09	-	
A3	0.11	0.47	3.44	-	
A4	-	0.35	2.70	1.08	
A5	-	0.25	1.31	1.07	
A6	-	0.13	0.62	0.87	mm/yr
B1	0.18	0.18	7.21	-	
B2	0.22	0.15	0.77	-	
B3	0.36	0.30	0.70	-	
B4	-	0.32	0.93	1.83	
B5	-	0.28	0.83	1.04	
B6	-	0.16	0.37	0.88	

TABLE IV
CORROSION PROPERTIES (CORROSION CURRENT)

Steels	NaCl 0%	NaCl 0.1%	NaCl 1%	NaCl 3%	I_{cor2} (A/Cm ² X 10 ⁵)
A1	4	7	7	-	
A2	1	3	27	-	
A3	1	5	44	-	
A4	-	3	9	9	
A5	-	2	7	9	
A6	-	1	5	7	(A/Cm ² X 10 ⁵)
B1	2	2	93	-	
B2	2	1	1	-	
B3	4	3	9	-	
B4	-	2	8	9	
B5	-	2	7	7	
B6	-	2	3	0.1	

C. Nondestructive Characteristic

The variation of the longitudinal ultrasonic velocities in frequencies of 5 and 10MHz, and transversal ultrasonic velocity in frequency of 5MHz is obtained for the samples. According to the obtained results for the longitudinal and transversal ultrasonic velocities' values, for 5MHz, it can be noted a higher velocity for the ferrite (5930 ± 3 and 3230 ± 1) and lower for the martensite (5884 ± 1) and (3180 ± 1.2), being the pearlite (5917 ± 2.5 and 3234 ± 2) with an intermediate value. The lowest sound velocity in martensite can be explained by high amount of tetragonal lattice distortion. The tensile properties as shown in Table V of A₂ samples are almost comparable to A₁. This may be due to the presence of some amount of tempered bainite and retain austenite, which improve the strength and ductility in mostly ferrite matrix. Further increase in hardness due to increase in acicularity microstructure. The mechanical properties study of the B₂ and B₃ reveals a bit of dissimilarity in characteristics to that of steel sample A₁, A₂.

TABLE V
MECHANICAL PROPERTIES

Steels	UTS (MPa)	VHN	Work hardening rate (n)	Elongation (%)
A1	456	169	0.323	44
A2	466	151	0.608	41
A3	576	191	0.612	42
B1	498	217	0.418	40
B2	576	179	0.421	32
B3	579	232	0.533	33

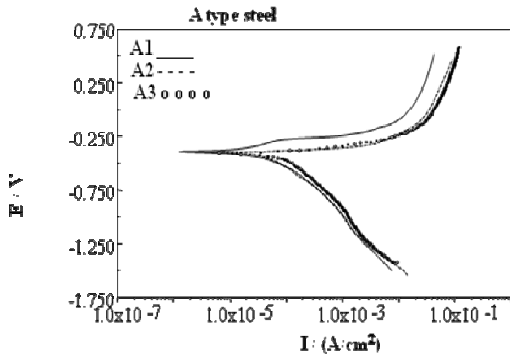


Fig. 8 Polarization plot for Steel A in 0.1% NaCl

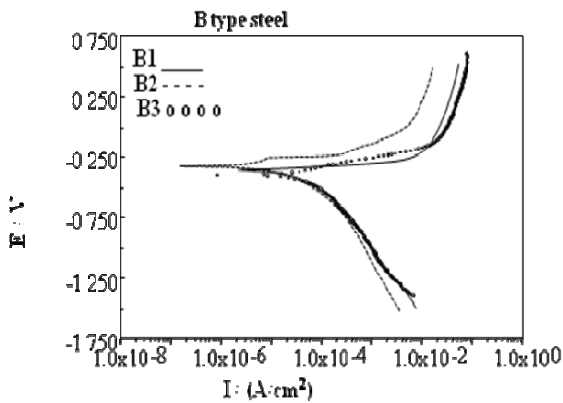


Fig. 9 Polarization plot for Steel B in 0.1% NaCl

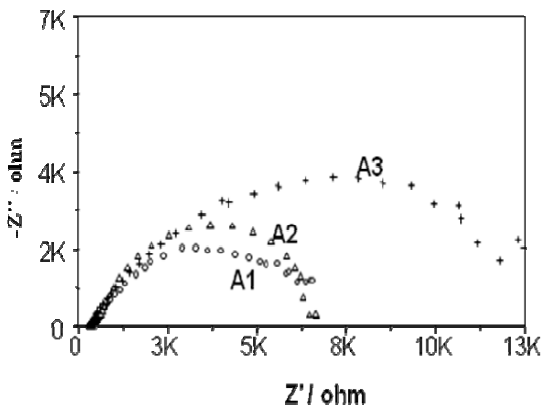


Fig. 10 Nyquist Plot of Steel A in 0.1% NaCl solution

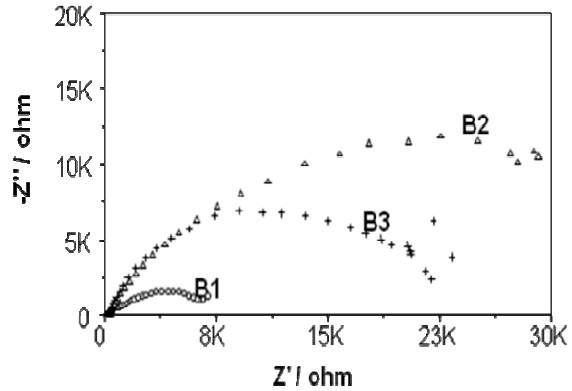


Fig. 11 Nyquist Plot of Steel B in 0.1% NaCl solution

IV. CONCLUSION

According to the results obtained, one can conclude the following. The highest ultrasound propagation velocity is observed for ferrite, then for pearlite and finally for martensite, both for longitudinal and transverse waves. Microstructure contains more low temperature transformation products like bainite and/or martensite and other non equilibrium are more corrosion. This behavior was observed in all frequencies used. Ultrasound velocity is mainly affected by the changes in the elastic module of the individual grains that is dependent upon the degree of lattice distortion and disorientation in the prior austenite grains. Steels in rolled condition are more corrosive in both the case, probably due to high dislocation density. The lowest ultrasound propagation velocity verified in martensite is due to the large amount of tetragonal distortion of the lattice that increases the elastic anisotropy of grains. Steel containing more Cu, Ni and Cr is less corrosive compared to other steels having same processing or microstructure.

ACKNOWLEDGMENT

The results presented in this paper were obtained within the project work of Master thesis in School of materials science and engineering, BESU, shibpur, west Bengal.

The authors would like to thank, Prof. N. R. Bandhyopadhyay with all the faculty members of school of materials science and engineering and Prof. (Dr.) Jayati Datta, dept. of chemistry, BESU, Shibpur for fruitful help for this project.

REFERENCES

- [1] G. Frommeyer and, O. Grassel: High strength TRIP/TWIP super plastic steels: Development, properties, Application, La Revue De, Metallurgie CIT, october, 1998, 1299.
- [2] Ultra light steel auto body-advanced vehicle technology (ULSABAVC) overview report, IISI, 2002, p.66, www.worldautoteel.org.
- [3] V. F. Zackay, E. R. Parker, D. Fahr and R. Busch: Trans. ASM, 60 (1967), p. 252.
- [4] Krüger L, Meyer LW, Brüx U, Frommeyer G & Grassel O Stress-deformation behavior of high manganese (Al, Si) TRIP and TWIP steels. J Phys IV 110: 189-194(2003).

- [5] Frommeyer G, Brüx U & Neumann P (2003) Supra-Ductile and High-Strength Manganese-TRIP/TWIP Steels for High Energy Absorption Purposes. *ISIJ Int* 43:438-446.
- [6] Han YS & Hong SH (1997) the effect of Al on mechanical properties and microstructures of Fe₃₂Mn-12Cr-xAl-0.4C cryogenic alloys. *Mater Sci Eng A* 222:76-83.
- [7] Matsumura, O., Sakuma, Y. and Takechi, H. *Transactions of the Iron and Steel Institute* 27 (1987) 570.
- [8] J. R. Challande (M. S. Thesis) UCRL-18475, (1968).
- [9] F. J. Padilla (M. S. Thesis) UCRL-19678, (1970). P.J. JAQUES, J. Ladriere, F. Delannay, *Metall.Trans.*32A, (2001); p.2759.
- [10] Khoshnaw F.M., Kheder A.I., Ali F.S.M. (2007): Corrosion behaviour of nitride low alloy steel in chloride solution, *Anti-Corrosion Methods and Materials*, 54/3, pp.173-179. doi: 10.1108/000355907107486441.
- [11] Chen Y.Y., Tzeng H.J., Wei L.I., Shih H.C. (2005): Mechanical properties and corrosion resistance of low-alloy steels in atmospheric conditions containing chloride, *Materials Science & Engineering A*, 398, pp. 47-59. doi:10.1016/j.msea.2005.02.064.
- [12] Turn ball A., Zhou S. (2008): Impact of temperature excursion on stress corrosion cracking of stainless steels in chloride solution, *Corrosion Science*, Vol. 50, pp. 913-917. doi:10.1016/j.corsci.2008.01.020.
- [13] Sadhukhan S., Das K. P., Bandyopadhyay N. R. and Banerjee M. K. *Journal of the Institution of Engineers, India*, 82 (2001) 65.
- [14] Bhadeshia, H. K. D. H. and Honeycomb, R. W. K. *Steels: Microstructure and Properties*, Elsevier, UK, (2006).
- [15] Takechi, H., Matsumura, O., Sakuma, Y. *Japan Kokai Tokyo Koho Japan Patent* 62,188,729 (1987).
- [16] Matsumura, O., Sakuma, Y. and Takechi, H. *Transactions of the Iron and Steel Institute* 27 (1987) 570
- [17] Kim, S. A. and Johnson, W. L., 2007, "Elastic constants and internal friction of martensitic steel, ferritic-pearlitic steel, and α -iron", *Material Science and Engineering A*, 452-453, pp 633-639.
- [18] Hakan, C.G. and Orkun, B.T., 2005, "Characterization of micro structural phases of steels by sound velocity measurement", *Materials Characterization* Vol. 55, pp. 160-166.
- [19] R. Prasad, S. Kumar, *Journal of Materials Processing and Technology*. 42 (1994) 51-59.
- [20] C.H. Gür, B.O. Tuncer, *Materials Characterization*. 55 (2005) 160-166.
- [21] V.H.C. de Albuquerque, T.A.A. Melo, D.F. de Oliveira, R.M. Gomes, J.M.R.S. Tavares, *Materials & Design*, DOI:10.1016/j.matdes.2010.02.010
- [22] P. Palanichamy, A. Joseph, T. Jayakumar, *Baldev Raj, NDT & E International*. 28 (1995) 179-185.
- [23] S.A. Kim, W.L. Johnson, *Materials Science and Engineering: A*. 452-453 (2007) 633-639.

Animesh Talapatra, received a B.E/B.TECH degree in Mechanical engineering from NIT, Agartala (Formerly Tripura engineering college) and a M.E/M.TECH degree in specialization Materials engineering from BESU, Shibpur, West Bengal, India. He has many teaching, research and industrial experience in the engineering (NDT field). Recently he is an assistant professor of MCKVIE (under west Bengal University of Technology, India) in automobile engineering department and connected as regular author, editor of various Nondestructive testing journals, Magazines. He has received many prestigious scholarships (GATE-2002,SRF) in India to do research and study in Indian Institute of Technology(IIT). He has published number of articles, papers in National, International journals and Magazines. He is related with as core member of various national and international conference, Seminar and workshop. He has completed various prestigious projects as a senior research fellow in field of Nondestructive testing.