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Prof. V S Raja, Emeritus Fellow in the Dept. of Met. Engg. and Mat. Sc., IIT Bombay delivered Prof. N. P. Gandhi Memorial Lecture 2023 at IIM-ATM 2023 held at KIIT Bhubaneswar

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Technical Article

Optimization of Austenitizing Temperature to Achieve Desired Properties of Heavy Section Austempered Ductile Iron (ADI) Casting

RKP Singh*, Vinayak Pawar*, Amol Gujar*, Shrikant Jadhav*, Dipak Tarade*

Abstract

In present study, the effect of the austenitizing temperature on microstructure and mechanical properties along with sub-zero temperature (-20°C) un-notched impact properties of samples made from 50 mm austempered ductile iron (ADI), Y blocks were studied. The specimens were austenitized at 850°C, 870°C and 890°C for 180 minutes. then austempered for 180 minutes. at 370°C, followed by air cool. The microstructural changes, tensile properties and sub-zero temperature un-notched impact properties examined. Volume fraction of retained austenite (%RA) measured by XRD analysis. The microstructural features at different heat treatment conditions were correlated with kinetics of transformation and mechanical properties.

Microstructure of samples austenitized at 850°C & 870°C shows mix structure of ausferrite, pearlite and pro eutectoid ferrite in core and surface region. Microstructure of samples austenitized at 890°C contains fully ausferrite structure in core and surface region. Testing results reveals that mechanical properties, ductility and un-notched impact energy were higher at 890°C. Y blocks austenitized at 850°C and 870°C shows lower mechanical properties and subzero un-notch impact energy. This was due to, at austenitizing temperature 850°C and 870°C sufficient carbon was not dissolved in matrix to suppress the pearlite formation in the microstructure. Percentage of retained austenite was higher for 890°C austenitization temperature due to comparatively high carbon dissolution in matrix. As volume fraction of retained austenite increases impact strength increases. Optimum mechanical properties were obtained for specimens austenitized at 890°C for 180 minutes and austempered at 370°C for 180 minutes. The outcome of this study

shows that the austenitizing temperature has a very significant impact on structure homogeneity and the resultant mechanical properties.

Keywords - ADI, Austenitizing temperature, Austempering temperature, %RA, Un-notched impact energy.

I. Introduction

Austempered Ductile Iron (ADI) provides the optimum combination of low cost, design flexibility, good machinability, high strength to weight ratio, good toughness, wear resistance, and fatigue strength to the design engineer. The extraordinary capabilities of ADI are attributable to its distinct microstructure, which consists of high carbon austenite (γ -HC) and ferrite (α). The austempering process was invented in the early 1930s as a consequence of research on the isothermal transformation of steel by Bain et al [1]. Flinn used this heat treatment on cast iron, specifically gray iron, in the early 1940s. Both the material, ductile iron, and the austempering technique had been invented in the 1950s. However, the technology for producing ADI on a large scale lags behind. Before extremely efficient semi continuous and batch austempering methods were invented and commercially applied to ductile iron, the 1970s would arrive [2]. ASTM A897-90 and ASTM A897M-90 requirements for austempered ductile iron castings were published in the United States by the 1990s, while other requirements were produced globally. In the recent two decades, applications for heavy section austempered ductile iron (ADI) castings have increased dramatically. Unfortunately, as the section size of the casting rises, so does the difficulty of obtaining desirable microstructures [3]. For example, increasing the section size slows down the cooling process, resulting in a low nodule count [4].

* Kalyani Center for Technology and Innovation, Bharat Forge Ltd, Pune 411036, Maharashtra, India



Micro shrinkage porosities, graphite degeneration, eutectic carbide production, and alloying element segregation can all occur in heavy section cast ductile iron. Heavy section ADI casting, on the other hand, requires a substantial amount of alloying materials to give appropriate austemperability in order to fully austemper the entire casting [5,6]. The greater the amount of alloving elements present, the greater the possibility of segregation of these elements. As a result, it is critical to employ appropriate and optimum amounts of the alloving elements to achieve the desired austemperability while also eliminating or minimizing element segregation [7]. When ductile or nodular cast iron is subjected to austempering heat treatment, a microstructure comprising ferrite (α) and high carbon austenite $(\gamma$ -HC) is formed. The resulting microstructure of austempered steel is a fine dispersion of carbide in a ferritic matrix known as bainite. The presence of a considerable amount of silicon in ductile cast iron prevents carbide formation. Because of this distinction, the austempered structure of ductile cast iron is frequently referred to as "ausferrite" rather than bainite. When ferrite occurs within the austenite during the austempering process of nodular or ductile cast iron, the carbon is rejected from these places and dissolves in the surrounding austenite. The carbon content of the austenite increases as more ferrite forms. Because the carbon content of this austenite is very high (in excess of 1.0%), it is stable at room temperature, and therefore, the final microstructure comprises of ferrite and high carbon and stable austenite. This is the desirable microstructure in ADI. However, if the austempering reaction is carried out for too long, a stage II or second reaction occurs, during which the high carbon austenite (γ -HC) might further decompose into ferrite and ε -carbide. This reaction is unfavorable due to carbide embrittlement [8]. As a result, the best mechanical properties of ADI are attained after the first reaction but before the second reaction begins. The process window is the time gap between the completion of the first reaction

and the start of the second reaction. The addition of alloying elements such as nickel and molybdenum can broaden the process window. As a result, typical ADI contains nickel (1.5%) and molybdenum (0.3%). The austempering temperature and time affect the microstructure of ADI. The shape of ferrite, preserved austenite content, carbon content of austenite, and presence or absence of carbides in austenite or ferrite are essential microstructural properties. Several researchers have previously investigated the effect of heat treatment parameters on the fracture toughness of ADI [9-13]. However, majority of these experiments have been conducted on ADI of conventional composition, that is, ductile cast iron having alloving elements such as nickel and molybdenum, as well as a relatively high manganese concentration (Mn=0.4%). Recent research has revealed that alloying elements like manganese and molybdenum increase segregation in ADI [14]. These segregated zones absorb lot of carbon, and during austempering, they can turn into martensite, which has poor mechanical properties. As a result of the austempering heat treatment procedure, ductile cast iron with no alloying elements and low manganese content may provide higher mechanical properties. In the literature, little work has been reported on sub-zero impact properties of austempered ductile iron specially for heavy section thickness.

The main aim of present study was to study, the effect of austenitization temperature on microstructure, mechanical properties, sub-zero impact properties, hardness and retained austenite percentage of austempered ductile iron 50mm thick Y block test coupons.

II. Experimental work

Material: The composition of the ductile iron used in this study is shown in table 1 was produced in medium frequency induction furnace in commercial foundry. The ductile iron was cast in the shape of 50 mm Y-blocks as per standard EN 1564. shown in Fig.1.

Table 1 : Chemical Composition of investigated ADI											
Element	C	Si	Mn	Р	S	Ni	Cr	Мо	Cu	Mg	CE
Actual	3.6	2.6	0.2	0.023	0.004	1.0	0.020	0.12	0.70	0.041	4.46



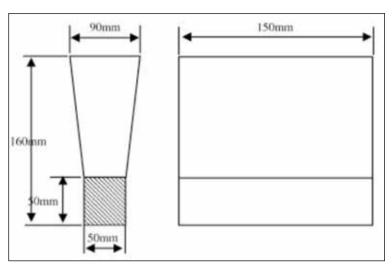


Fig. 1 : Y block as per standard EN 1564

Heat treatment : Y-blocks were austenitized at 850°C, 870°C and 890°C for 180min and transferred rapidly to a salt bath held at predefined austempering temperature of 370°C for 180min. All testing specimens were machined from bottom section of Y-block in order to avoid elemental segregation effects and porosity.

Tensile testing : Tensile tests were performed in a servo-hydraulic MTS test machine at a constant loading rate of 40 lb./s. By connecting an X-Y plotter to the test equipment, an autograph record of the load versus displacement was obtained from each sample. Tensile testing was performed at room temperature and ambient atmosphere according to ASTM standard E-8 [15]. At least three identical test samples were evaluated for each heat treatment cycle, and the average values from these samples were used as the test data representative.

Charpy impact testing : At least four un-notched impact samples measuring $10 \times 10 \times 55$ mm were prepared from each heat treated specimen. Impact test were performed in accordance with EN ISO 148-1. In order to avoid error in results due material defects like porosity and elemental segregation, higher three values used as testing representative. Samples were examined at a -20°C by maintaining temperature with ethanol + liquid nitrogen.

X-ray diffraction: X-ray diffraction analysis was carried out to estimate the retained austenite content in specimen. It was done using a monochromatic copper K α radiation at 30 KV and 10 mA. A Rigaku

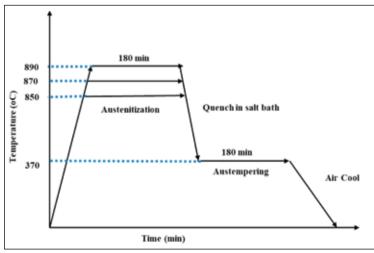


Fig. 2 : Heat treatment cycle



• Materials Engineering

rotating head anode diffractometer was used to scan the angular 2u range of $40-50^{\circ}$ at a scanning speed of 0.05° per sec. The profiles were analyzed on a computer to obtain the peak positions and the integrated intensities of (111) plane of austenite and (110) plane of BCC ferrite. The volume fraction of ferrite and austenite can be estimated [16] as follows:

Va + Vf =100%

Where, Va = volume fraction of retained austenite Vf = volume fraction of ferrite.

Metallography : Samples have been ground, polished and etched with 3% nital solution following the standard metallography procedure for microstructural examination. Microstructure of the heat treated samples were examined by Carl Zeiss optical microscope after etching.

Hardness testing : Hardness of heat treated specimens were checked by Brinell hardness tester of dia. 10 mm ball indenter with 3000kgf load.

III. Results and discussion

Microstructural changes after different austenitizing treatments:

As cast microstructure shows graphite nodule surrounded by ferrite, typical "bulls eye" structure, in perlitic matrix as shown in Fig. 3. The morphology of the matrix after austempering depends on the austenitizing temperature and austempering time.

Fig. 4 (a-f) shows the microstructure, at surface (a. c. e) and core (b. d. f) for the specimens austenitized at 850°C, 870°C, & 890°C for 180 min and austempered at 370°C for 180 min respectively. Y-block austenitized at 850°C shows mixed microstructure of ausferrite and pearlite along with graphite nodule at surface, while pearlite and pro-eutectoid ferrite with graphite nodule at core. Y-block austenitized at 870°C shows fully ausferrite structure with graphite nodule at surface and at the core ausferrite and traces of pearlite around graphite nodules are observed. The pro-eutectoid ferrite occurs as a result of a low austenitizing temperature, i.e. as a result of austenitizing the iron in the austenite-ferrite-graphite region of the phase diagram (Fig. 5) [17]. Fig.5 shows that austenitizing temperature at 850°C is insufficient for full austenitization. Microstructure containing this type of ferrite have low tensile strength as compared those with fully ausferritic structure. Y-block austenitized at 890°C temperature shows fully ausferrite structure at surface and core region. From the micrographs it's evident that with increasing austenitizing temperature, hardenability of ADI is increasing which subsequently suppress pearlite formation and increase in ausferrite content.

During the austempering process of the cast samples, the perlitic matrix transforms to austenite and a little quantity of ferrite in the region where the two phases ferrite and austenite coexist, which

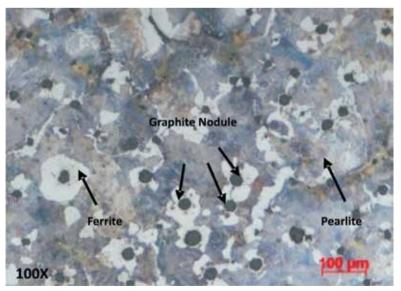


Fig. 3 : As cast Y block microstructure



is still controlled by the austenitizing temperature. The austenite that has accumulated at the ferrite grain boundaries begins to expand through the ferrite grains. The creation of ferritic matrix layered with carbon-enriched austenite is the result of such growth. Further nucleation of the austenite at the grain boundaries of the primary ferrite results in carbon enrichment and saturation of the austenite, but subsequent carbon diffusion becomes complex due to the termination of ferrite grain development.

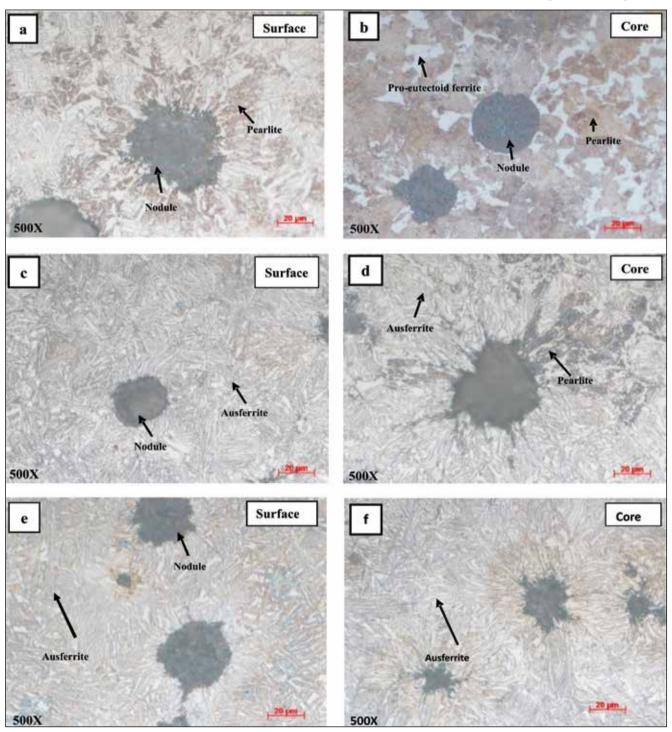


Fig. 4 : Microstructure after austempering at surface (a, c, e) & core (b, e, f) of samples austenitized at 850°C, 870°C & 890°C resp.



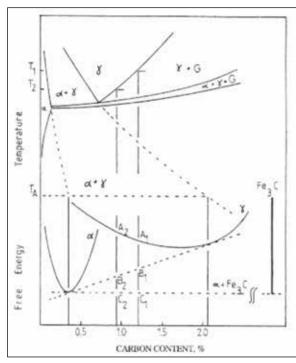


Fig. 5 : Schematic Fe-C-Si phase diagram and free energy curves for ferrite (α), austenite (γ) and cementite (Fe₃C) phases

Fig. 6 shows effect of austenitizing temperature on sub-zero (20°C) impact properties at surface (L1, L4) and at core region (L2, L3) region. As fully ausferritic structure was developed at 890°C austenitizing temperature, impact properties are higher as compared to Y-blocks austenitized at 850°C and 870°C. Y blocks austenitized at 850°C and 870°C shows mixed microstructure of ausferrite, pro-eutectoid ferrite and pearlite. Even the trace fraction of pearlite can significantly reduce the impact strength.

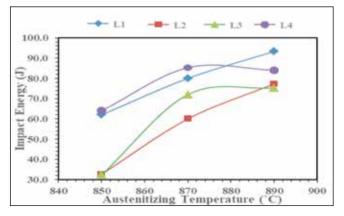


Fig. 6 : Impact vs Austenitizing temp.

Fig. 7 shows effect of austenitizing temperature on mechanical properties. As austenitizing temperature is increased from 850°C to 890°C, the carbon content in matrix increases which leads to increase in yield strength (YS) and ultimate tensile strength (UTS). But slight drop in elongation is observed at 890°C austenitizing temperature due to slight coarsening of ferrite needles.

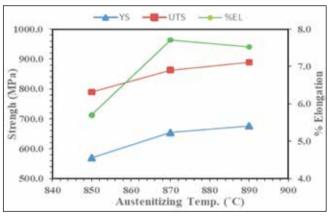


Fig. 7 : Mechanical properties vs Austenitizing temp.

Fig. 8 shows the hardness variation at surface and core for the specimens austenitized at three different temperatures. Results shows that at 850°C and 870°C austenitization temperature, variation of hardness from surface to core is more due to different microstructure at which consists of ausferrite at surface and pro-eutectoid ferrite, pearlite at core region. Austenitization at 890°C temperature hardness variation from surface to core is very less. As fully ausferritic microstructure is observed throughout sample from surface to core. At 890°C austenitization, hardenability of material is higher due to high carbon in the solution which increases the depth of austempering.

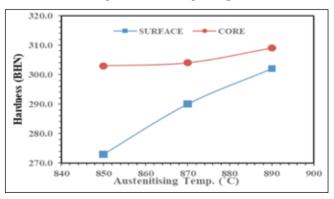


Fig. 8 : Hardness vs Austenitizing temp.





Metallurgy

Fig.9 shows the results of the austenite fraction calculations by comparison method as a function of the austenitizing temperature at core region of samples. From fig. 9, it follows that the austenitizing temperature has a significant effect on the highcarbon austenite fraction. An increase in the austenitization temperature raises the austenite fraction in the microstructure. This is the result of a higher carbon content in the austenite with a rise in the austenitization temperature. At austenitization temperature of 890°C volume fraction of retained austenite is higher as compared to samples austenitized at 850°C and 870°C. At 890°C austenitization temperature higher carbon got dissolved in austenite matrix which promoted high retained austenite stabilization during austempering heat treatment process. As volume fraction of retained austenite increases impact strength increases as shown in Fig.10. Retained austenite between ferrite laths suppress micro cracks initiation and hinder the propagation of cracks in ADI cast iron which leads to higher impact properties at 890°C austenitization temperature. The XRD patterns of all samples investigated are presented in Fig.11. Small austenite peaks were visible in the XRD patterns, which also supported the formation of high carbon retained austenite in all samples. High carbon enriched austenite is very stable at room temperature. XRD plots clearly shows difference of austenite peak (γ -111), Austenitization at 890°C gives taller and broader peak as compared to samples austenitized at 850°C and 870°C.

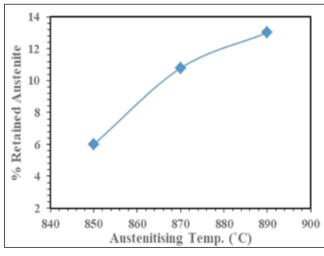


Fig. 9: % Retained austenite vs Austenitizing temp.

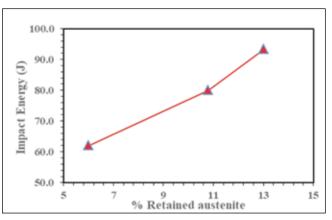


Fig. 10 : Impact energy vs % Retained austenite.

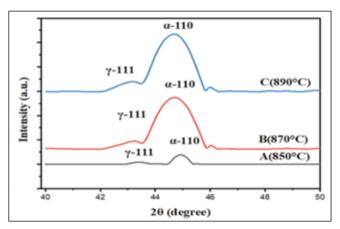


Fig. 11 : XRD plot of samples austenitized at 850°C, 870°C and 890°C

IV. Conclusion

- The Austenitizing temperature has significant effect on the transformation rate during the subsequent austempering process
- Heat treatment at 850°C resulted in pro-eutectoid ferrite and pearlite in microstructure.
- This is attributed in lowering Austenitizing temperature which reduced stability of carbon in austenite.
- At 870°C Austenitizing temperature, traces of pearlite observed in microstructure while 890°C Austenitizing temperature resulted fully ausferrite structure.
- As austenitizing temperature increases unnotch impact energy, yield strength and UTS also increases. Even the trace fraction of pearlite can significantly reduce the impact strength.



- Retained austenite volume fraction increased with increasing austenitizing temperature due to high carbon goes in to solution which results in stabilization of austenite. Maximum retained austenite obtained at 890°C austenitization. As volume fraction of retained austenite increases impact strength increases.
- The outcome of this study shows that the austenitizing temperature has a very significant impact on structure homogeneity and the resultant mechanical properties.
- Optimum mechanical properties achieved at austenitization 890°C for 180 min and austempering at 370°C for 180 min.

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AUTHORS

First Author – Dr. RKP Singh, Senior Director, KCTI, RajkumarSingh@bharatforge.com Second Author – Mr. Shrikant Jadhav, M.Tech. Dy. Mgr. (KCTI) Shrikant.jadhav@bharatforge.c Third Author – Mr. Amol Gujar, M.Tech, Mgr. (KCTI), Amol.gujar@bharatforge.com. Fourth Author – Mr. Dipak Tarade, M.Tech, Asst. Mgr. (KCTI) Dipak.tarade@bharatforge.com Correspondence Author – Mr. Vinayak Pawar, M.Tech. Sr. Mgr. (KCTI) Vinayak.Pawar@Bharatforge.com



Prof. N. P. Gandhi Memorial Lecture 2023 Technologies and Fin

Basics, Innovations and Embracing Challenges towards Addressing Corrosion in Critical Technologies and Environmental Concerns



V S Raja

Department of Metallurgical Engineering and Materials Science Indian Institute of Technology Bombay, Mumbai 400 076, INDIA Email: *vsraja@iitb.ac.in*

Abstract

The subject Corrosion Science and Engineering, by nature, has always been at the forefront of protecting environment even when advanced technologies developed in the Industrial era may not take cognizance of this issue. As all the engineering metals are deemed to suffer corrosion dictated by their thermodynamic, it is imperative to develop these engineering materials to offer reliable and long-lasting service life. Engineering these materials to offer kinetic resistance to corrosion thus forms the basis of materials development. This involves understanding interplay of metallurgy, chemical environments and the operating conditions such as applied stresses. The modern technologies demanding lighter and smart structures, ability for the materials to withstand high temperatures and pressures and being environmentally friendly pose interesting challenges to corrosion researchers to be innovative, understand the basic science of corrosion and also design difficult experiments to simulate the operating conditions. This talk will touch upon a few studies that are of interest to indigenization of robust and environmentally friendly technologies carried out in our laboratory.

Keywords : Al-alloys, environmentally assisted cracking, protective coatings, AUSC oxidation.

1. Introduction

Metallic materials are widely employed as structural materials to build infrastructure, industrial plants and transportation systems and as well as in manufacturing devices to meet various needs of human kind. A closer look at the metallic elements employed for structural applications and the majority of those used to manufacture devices are dictated by thermodynamics that they suffer corrosion in chemical environments of various kinds. The threat to corrosion induced failure of structures increases with the need to apply them in more stringent conditions such as deep-sea exploration of resources and light weighting structures and Advanced Ultra Super Critical Power Plants (AUSC). These technologies are vital as the resources in earth crest deplete and the global warming due to carbon dioxide emission possess a greater threat to the human kind. In addition, advanced manufacturing technologies such as additive manufacturing are expected to alter the corrosion behavior of metallic materials.

As suggested earlier, the engineering alloys have the inherent thermodynamic tendency to corrode which cannot be changed. The successful application of alloys to manufacture reliable, safe and long-lasting structures, therefore, hinges on importing kinetic resistance to the corrosion of these alloys. This essentially means understanding the underlying corrosion mechanisms. These mechanisms vary significantly and can become complex as the alloys are made into structures and devices to perform different function (for examples corrosion related to pipelines, pressure vessels, welded and unwelded structures, electronic devices differ significantly) Corrosion in aqueous environments is primarily governed by electrochemical phenomena. These phenomena, is greatly influenced by the metallurgy of the alloy at microscopic and atomic level while the role of environment in altering electrochemical phenomena needs no emphasis, the mechanical aspect of structure in change the corrosion mechanism is very important. Thus, a larger goal in developing alloys having higher corrosion



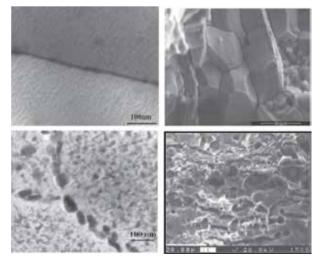
resistance must involve understanding the inter play of electrochemistry, metallurgy and mechanical. The core objective of corrosion research in our group has been to address these aspects for alloys ranging from stainless steels, aluminum alloy, titanium allovs, nickel base allovs, iron based intermetallic and maraging steels towards this goal. As protective coatings are the most widely employed technology to mitigate corrosion, especially where the alloys meet the mechanical requirements but fail to offer corrosion resistance (steels for atmospheric applications and nickel base alloys for high temperature applications), research towards novel and environmentally friendly coatings was directed. Notably, our research emphasis has been towards materials and technologies that are developed in our country and yet focus on understanding the corrosion mechanisms. In that direction, we have developed indigenous facilities to study the oxidation performance of nickel base alloys and stainless steels in steam at Advanced Ultra Super Critical (AUSC) conditions (710°C and 32 MPa), a step towards realizing such thermal power plants. In this lecture I shall present briefly some of our work related to (1) Development of high strength and high environmentally assisted cracking (EAC) resistant aluminum alloy, (2) Development of novel and eco-friendly coating and (3) some challenges in establishing test facilities to study oxidation behavior of metallic materials for AUSC applications.

2. Development of high strength and high environmentally assisted cracking resistant Alalloys:

2.1 Role of grains boundary and matrix precipitates.

This work started almost two decades back when Dr. A.K. Mukhopadhyay was indigenizing 7010 Al-alloy. The primary aim was to develop heat treatment other than the conventional over aging (OA) and industrially unsuitable retrograde raging treatment (RRA) to increase strength and resistance to EAC. This was done by aging below G.P. solvus line to promote homogenous nucleation and then subject the alloy to peak and over aging (1). As it turned out, the peak-aged alloy tested in 3.5 wt.% NaCl at a strain rate of 1×10⁻⁶ having $\sigma_{\rm uts}$ = 575 MPa and ε =12% in air suffered intergranular showing with $\sigma_{\rm uts}$ = 575 MPa and ϵ = 3%. Thus, a remarkable 66% reduction in ductility for the alloy in the environment was noticed. On the contrary, the overaged alloy showed σ_{uts} = 560 MPa and ϵ = 13% in air and in the environment, it showed σ_{uts} = 560 MPa and $\varepsilon = 12\%$ almost retaining its ductility shown in the air. Notably, the over aged alloy lost about 15% of its strength (Figure 1 a-b). A detailed investigation lead to our belief that, the intergranular cracking was mainly associated with a relatively more anodic character of the grain boundary precipitates (GBPs) namely MgZn₂(Cu). Upon over aging, these GBPs turned relatively noble due to enrichment of Cu (Figure 1c). Through detailed analysis, we proposed the preferential dissolution of the GBPs is the main reason, which needs to be suppressed in order to promote EAC resistance to the alloy.

This happens when the copper content of the GBPs is increased to make the latter noble. This



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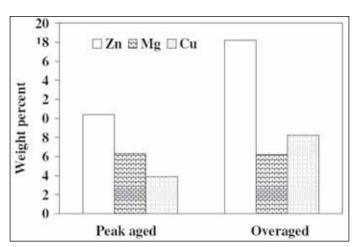


Fig. 1 : Top figures for peak aged 7010 Al-alloy. Left: TEM image of the alloy and Right depicts intergranular fracture occurred in SSR tested specimen. Middle figure for the overaged alloy. Left: TEM image of the alloy and Right depicts ductile transgranular fracture occurred in SSR tested specimen. The plot at the bottom is energy dispersive x-ray analysis data of grain boundary precipitates. Note a significant increase in the GBPs of the over aged alloy (ref.1)

mechanism of the active grain boundary precipitates causing EAC was first of its kind as hitherto EAC susceptibility of the alloys were considered to be due to the coherent precipitates in the overaged alloy has been suggested promote plan slip. On over aging, the precipitates turned incoherent which is expected promote cross-slip and hence retain the ductility of the alloy even in corrosive environments. This aspect will be discussed in details later.

2.2 Role of recrystallization:

The fact the GBPs in the peaked alloy is predominantly responsible for the EAC susceptibility of the alloy, lead to the conclusion that suppressing recrystallization can further explore the EAC resistance of the alloy. The base alloy AA 7010 has Zr which forms Al₂Zr

dispersoids. These dispersoids are expected to suppress recrystallization but are found be less effective. A.K. Mukhopadhvav increased added Sc to this alloy that formed $Al_3Sc_xZ_{1-x}$ precipitates are more effective in inhibiting recrystallization. Unsurprisingly the Sc added alloy showed remarkable EAC resistance even in the peak aged condition (Figure 2) (2). Thus, the Sc added alloy is air showed σ_{uts} = 560 MPa and ϵ = 13% and in 3.5 wt% NaCl tested using slow strain rate test at 1x10-6 strain rate showed $\sigma_{\rm uts}$ = 560 MPa and ϵ = 12%, which is remarkable as the alloy has high strength as well as high EAC resistance. However, considering Sc is a strategic element, this is not a viable technology to produce Al-alloys having high strength as well as high EAC resistance.

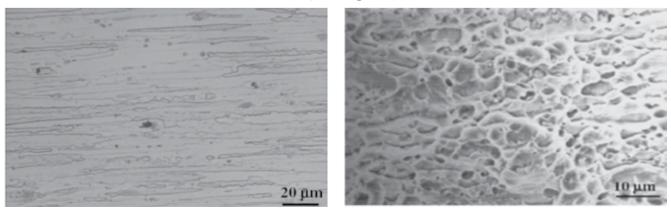


Fig. 2 : Left optical image of the Sc added 7010 Al-alloy and the right the fracture features of SSR tested alloy showing ductile features (ref.2)



2.3 Designing Alloys with noble GBPs and fine matrix precipitates.

The foregoing work has shown that so long as the GBPs are noble, the alloy will resist EAC. Therefore, it is possible to have fine (coherent) precipitates within the grains, the alloy is expected to resist EAC so long as the GBPS are noble in character. This way, it is possible to achieve Al-alloys with high strength as well as high EAC resistance. It is desirable that such a treatment can be employable in industries. The PhD research scholar Ajay Krishnan examined various possibility and came out with a modified T6I6 treatment, to shorten the aging time significantly and termed it as modified aging (3-5). The low-temperature aging treatment facilitated large five precipitates due to high under cooling as well a significant reduction in the solubility of Cu. Figure 3 compares the types of precipitates found in the peak aged, over aged and T6I6 aging conditions for 7010 Al-alloy (3). Higher population fine precipitates rendered high strength to the alloy and significantly high Cu content of GBPs (to the tune of about 6 wt.%) further resisted IGC. A twin objective of high strength and high EAC resistance could be achieved not only for 7010 Al-alloy but also for 7050 Al-alloy.

Figure 4 shows a typical slow strain rate curve for 7050 Al-allov tested in 3.5 wt.% NaCl at the 10⁻⁷ strain rate (4). As can be seen that MA alloy showed a remarkable 100 MPa higher tensile than the conventional over aged allow with even a marginal increase in the ductility of the alloy. Thus, the twin objectives of achieving high strength as well as high EAC resistance has been achieved. This work has been patented (5,6). While the above results are encouraging, fracture mechanics studies on notched specimens and hydrogen charging on smooth cylindrical specimens show that modified aged and over aged 7010 Al-alloy suggest that at high hydrogen concentrations, these alloy can be made susceptible to EAC even in these improved metallurgical conditions (7) Therefore, there is a need to understand more in depth the governing factors controlling EAC. This aspect is discussed in the next section.

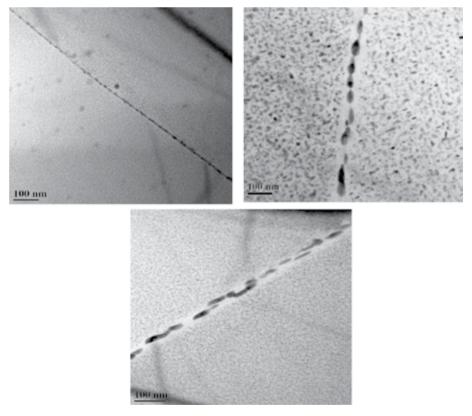


Fig. 3 : Comparison of TEM images of (a) peak aged, (b) overaged and (c) T6I6 aged 7010 Al-alloy (ref.3)



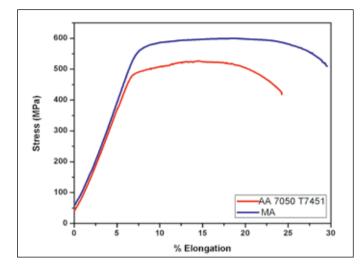


Fig. 4 : Slow strain rate test plots of AA 7050 obtained in 3.5 wt.% NaCl obtained at 1x10⁻⁷ strain rate (ref.4)

2.4 Deformation electrochemistry metallurgy in EAC of 7050 Al-alloy.

Environmentally assisted cracking susceptibility is evaluated using a slow strain rate test (SSRT). The loss in elongation for a specimen subjected to SSRT in a given environment in relation to the elongation shown by the alloy in air is considered as an indication of the extent of EAC susceptibility an alloy in a given environment. Rahul Kumar Agrawal, in his doctoral work has shown that work hardening is a better reflection of EAC susceptibility of 7004 Alalloy than elongation (8). Using the 7004 Al-alloy he shown that even though the alloy ductility does not get affected due to the environment (3.5 wt.% NaCl), its work hardening parameter significantly changes due to the environment. Later, he followed up this aspect by examining Geometric phase analysis, to determine the atomic strain in the alloy with and without exposure to environment at a given strain (9). A detailed study in this direction was further undertaken to understand Deformation-Electrochemistry-Metallurgy in EAC of 7050 Alallov by Shweta Shukla in her doctoral work. A brief account of this work is provided here. A more detailed account of her work can be found in our publications (10-12).

Several types of aging (T6, T73, T616, MA, RRA) are carried out to towards raising the mechanical behavior of Al-alloys. In each of these cases phases differing in nature and volume fraction are generated. The question is, can we understand

how these phases influence electrochemical and deformation behavior that ultimately decide the EAC tendency of Al-alloys. To address this question Shweta tailored heat treatments to generate variation in phases (quantitative or qualitative as it was not possible just to produce one kind of phases) (10) (Table). In all these cases the noble character of the GBPs precipitates was maintained to suppress the intergranular cracking behavior of the alloy.

Table 1 : Phases formed in 7050 Al-alloy							
Heat treatment	Matrix Precipitates size	Phases present					
OA	13 ± 4 nm	η' and η					
MA5	7.6 ± 2 nm	GP I, GPII, η' and η					
MA3	5.4 ± 1 nm	GPII and η'					

These had very interesting outcome which can be summarized as follows:

(1) The metastable phases GP II zone and GPI zone showed higher hydrogen kinetics $(2H_2O_{(1)}+2e^- \rightarrow H_{2(g)}+2OH_{(aq)}^-\& 2H^+_{(aq)}+2e^- \rightarrow H_{2(g)})$ and lower oxygen reduction kinetics $(O_{2(g)} + 2H_2O_{(1)} + 4e^- \rightarrow 4OH_{(aq)})$ in relation to the over aged alloy that contain η' (MgZn₂) matrix precipitates and η (MgZn₂) GBPs (Figure 5). It is known that hydrogen evolution reaction is beneficial towards EAC resistance of the alloy. Therefore, the above observations are rather surprising as alloys with GPII zones and





GPI zone are detrimental than η' and η from the electrochemical point of view and yet the alloy with these precipitates showed higher uniform elongation (less localization) and better strength and EAC resistance than OA alloy having η' as matrix precipitates.

(2) The work hardening θ_{max} and dynamic recovery rate $d\theta/d\sigma$, showed that hydrogen charging promotes cross-slip in the alloy with coherent precipitates, while it promotes planar ship in the alloy having semi coherent precipitates (Figure 6). The work also brings out the role of hydrogen and the nature of precipitates in generating dislocation and its effects on recovery rate. The precipitate

electrochemistry-dislocation interaction is presented schematically in Figure 7.

(3) The main outcome of the work is that there is still a large scope in improving the EAC resistance of copper containing 7xxx Al-alloy. Computational materials science can help to arrive at precipitates chemistry that will have lower hydrogen evolution kinetics and higher oxygen reduction kinetics without compromising on the coherent character of precipitates. Such modification in precipitates chemistry to suppress hydrogen chemistry is expected to show high strength along with high EAC resistance.

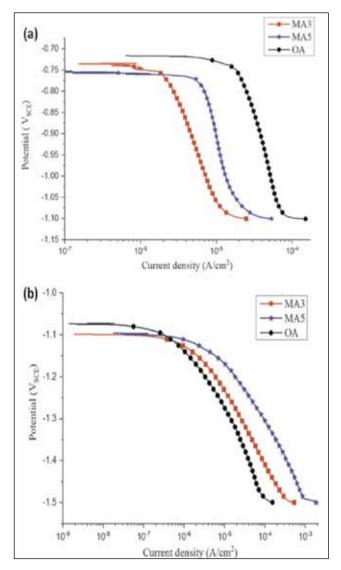


Fig. 5 : Polarisation curves of the OA, MA5 and MA3 in (a) freely exposed (b) deaerated 3.5 wt% NaCl environment (ref.10)



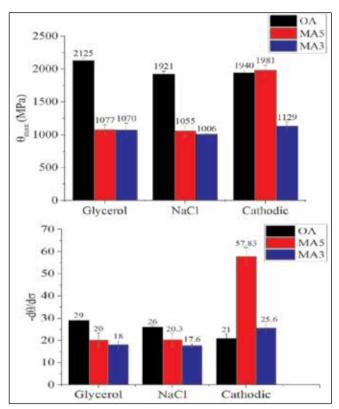


Fig. 6 : Work hardening parameter and dynamic recovery rate for 7050 Al-alloy under various testing conditions (ref.10)

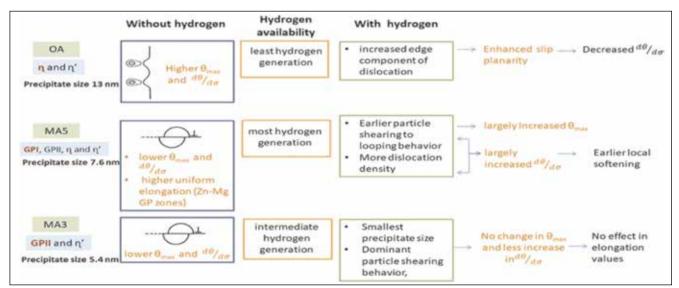


Fig. 7 : The finding of the work can be summarized schematically as above (ref.10)

3. Development of Innovative coatings for Corrosion Protection

Protective coatings are a one of the majorly employed technologies to protect metals against corrosion. Among the coatings, paints/organic coatings are the

most widely employed due to its versatility within the metallic coatings zinc applied through hot dip technique stands the most. The application of paints is fraught with pollution due to emission of volatile organic compounds (VOC), due to extensive use of



solvents, mostly organic in nature. Typically, these paints release 28.8 gm of volatile compounds in air per litre of paint employed.

As far as the zinc coatings are concerned with the raising cost of zinc, it is becoming very expensive and there is a need to reduce zinc coating thickness to enhance the service life of the zinc coating. Innovative means were applied to address these problems.

3.1. Development of thermally sprayable polyethylene coatings

This is the doctoral work of S.K. Singh, Naval Materials Research Laboratory and master's work of Rashmi David. Low-density polyethylene (LDPE) is quite less expensive. But it lacks adhesion to metals when applied as coating, as there are no polar groups in polyethylene. This was addressed by grafting the low-density polymer with maleic acid using a versatile technique called reactive extrusion. More details can be found in the publications (13-16). Extrusion gave the LDPE a polar character (Figure 8). The grafted LDPE was cryoground into fine powders and the coating was applied through flame spray technique. Optimization of the parameters are required to ensure that the coating does not disintegrate due to exposure to flame. The coating so obtained showed significantly higher adhesive strength from 5.4 MPa to 8.8 MPa due to maleic acid grafting. Typical Bode plot obtained on the coated panel exposed to simulated sea water is shown in Figure 9. As can be seen the coating showed an excellent impedance, meaning high resistance to corrosion even after long exposure to sea water.

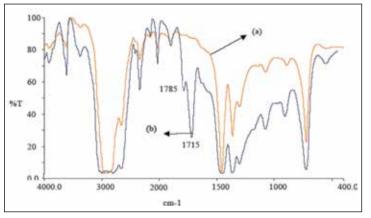


Fig. 8 : FTIR spectrum of (a) LDPE and (b) LDPE-g-MAc, 8% maleic acid (ref.14)

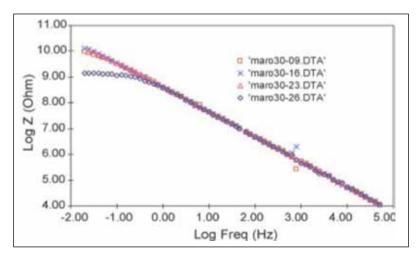


Fig. 9 : Bode plots of LDPE-g-MAc+ 0% red iron oxide after (\Box) 9 days; (×) 16 days; (Δ) 23 days; (◊) 26 days). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of the article (ref.14)



3.2 Development of high-performance hot dip galvanized coatings.

S.T. Vagge, for his doctoral work, carried out an interesting study on very conventionally used hot dip coatings. The objectives of the work are (a) to improve the formability of the coating (b) increase its corrosion protection capability. The main approach to address the problems is to refine the grain size of the zinc coatings strontium being an effect element to refine Al-Si coatings, it was employed in that study. He published series of papers exploring the influence of Sr on modification the grains and its effect on adhesion, formability and corrosion resistance (17-20). He also utilized simple polarization

technique to determine the coating delamination that might occur during sheet metal forming. The Sr addition has been found to show significant improvement in the corrosion resistance, adhesion strength and delamination resistance of hot dip zinc coating. Subsequently, with the help of coating simulator and Mahesh Walunj, we were finally able to demonstrate the significant beneficial effect of Sr addition on the corrosion resistance of Sr added coatings. For a coating thickness of ~10 µm addition of 0.02 wt.% Sr gave rise to a change in grain size from 593 µm to 145 µm (Figure 10) and salt spray life from h to h, thus demonstrating a form-fold improvement in corrosion performance (Figure 11) through adding trace amount of strontium.

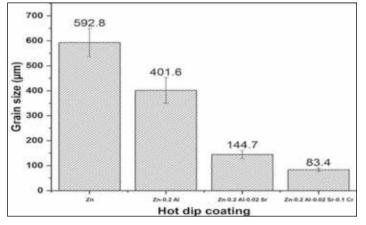


Fig. 10 : Comparison of grain size of various zinc coatings



Fig. 11 : Comparison salt fog exposed coated panel (a) plain zinc coating and (b) Zn-0.02Sr Coating.



4. Indigenous development of high temperature and high-pressure test loop and study of oxidation performance of Alloys for Advanced Ultra Super Critical Power Plants- A Challenging Task.

In the era of global warming increase in thermal efficiency of coal fired boilers to reduce carbon dioxide emission needs no emphasis. Advanced Ultra Super Critical (AUSC) power plants projected to operate around 710°C and 32 MPa is expected to offer thermal efficiency close to 50%. The stability of materials against corrosion in waterside at this temperature is critical for reliable and safe operation of power plants.

We have taken upon ourselves to establish a facility in the country to study oxidation behavior of potential alloys in AUSC environment. Figure 12 shows typical photos of various units. The flow diagram outlines the various associated units. Several alloys namely IN617, 740H, 304HCu and Sanicro 25 are under investigation. Here I bring out some salient results related to IN617, an alloy proposed to be employed in AUSC power plants.

In a recently published review by Ghule et al. (21), it has been brought out that only limited studies have been carried out on the oxidation behavior of IN617 alloy. It is hard to make definite conclusions on the behavior of the because of the fact that the water chemistry is either not specified or at too high oxygen levels. Therefore, a study was undertaken to simulate the water chemistry. The detailed work can be found in ref. 22. Typical kinetic curves of the alloy obtained at 710°C and 650°C with 32 MPa are shown in Figure 13 (22). Within the test duration of 600 h, the alloy showed less significant weight gain with near parabolic kinetics detailed mechanism of oxidation has been well brought out in the publication. The formed oxides were studied using Raman spectroscopy and transmission electron microscope.

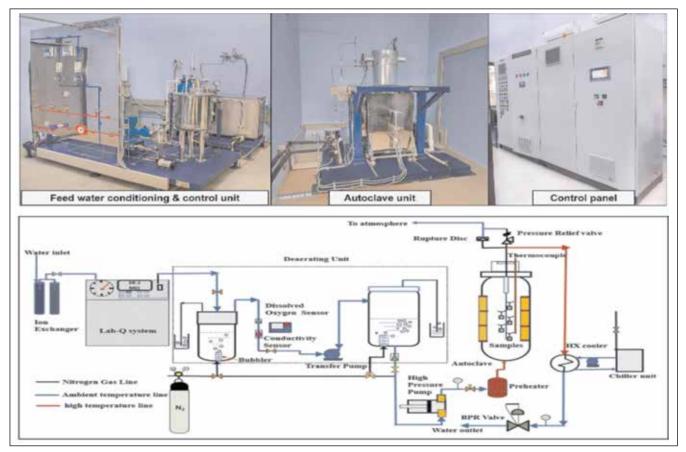


Fig. 12 : (a) Photos of the three major units of the AUSC test loop employed for the steam oxidation studies and (b) Schematic of the Advanced Ultra-Supercritical steam oxidation test facility (ref.22)



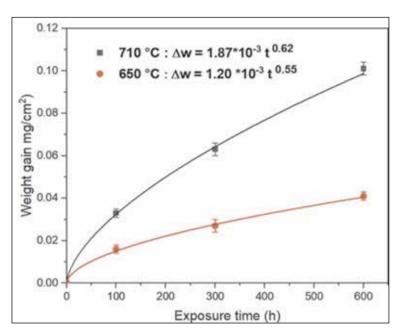


Fig. 13 : Oxidation kinetics of the alloy 617 in AUSC steam at 650 °C and 710 °C temperatures (ref.22)

The study revealed the oxidized alloy to have bilayer oxides, with the outer oxide being spinel of type MCr_2O_4 and the inner oxide being Cr_2O_3 (Figure 14). A notable outcome of the work is that no significant chromium volatilization has been observed in this alloy.

Metallurgy

5. Concluding Remarks

Corrosion is an important rate determining step in controlling the service life of engineering components. With the growing concerns of depletion of resources and deteriorating environment the emphasis on corrosion mitigation has gathered a great momentum. The modern technologies make the corrosion mitigation more complex. The way forward to address the corrosion issues is to understand the corrosion mechanisms followed by innovation to develop new materials and protective measures and take up challenging research as demanded by the advanced technologies. This talk and the paper highlight these aspects through the work carried out in our laboratory.

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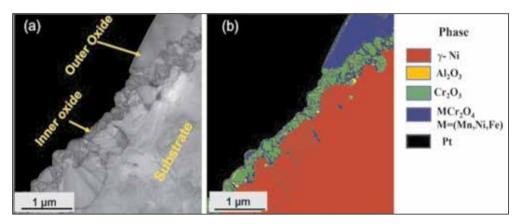


Fig. 14 : PED analysis and the resulting (a) Index map and (b) phase composition map of oxide scales formed on alloy 617 after 600 h in AUSC steam at 710 °C (ref.22).



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News Updates Domestic

SAIL wants to increase coking coal purchases from Russia

Steel Authority of India wants to increase coking coal purchases from Russia due to cheaper prices and is expecting four shipments, each with a capacity of 75,000 tons, in the quarter ending December, Chairman Amarendu Prakash said.

Coking coal, a key raw material in steelmaking, is in short supply and Russian coking coal is cheaper compared with the ones from Australia, Prakash told reporters on the sidelines of an industry conference.

Australia accounts for more than half of India's coking coal imports of around 70 million metric tons a year. India also imports coking coal from Russia and the United States.

The Economic Times (6.11.23)

Global market to soon see Made-In-India branded steel products: Scindia

The government along with the domestic steel industry will soon introduce Made-In-India branded steel products to the world market, according to Union Steel Minister Jyotiraditya Scindia.

This move will ensure standardised quality of products made by Indian steel producers, in addition to making domestic products more attractive to buyers, an official statement said. This is the first ever initiative by any ministry to introduce labelling and branding for products of a sector.

Scindia said the single brand identity for Indian-made steel will represent India's strong manufacturing potential. This branding has been rolled out for select products of all major Integrated Steel Producers (ISPs). All ISPs, and 65% of India's steel products have been on-boarded with common labels finalized for all the product categories. "Size and space for the Made in India logo has been allocated for each label. Made in India text will be used till the logo is finalized by Department for Promotion of Industry and Internal Trade (DPIIT)," he said.

The Economic Times (23.11.23)

India's steel demand to touch 190 Mt-mark in 2030; production to reach 210 Mt: SteelMint

India's steel demand is expected to grow at a CAGR of 7 per cent to touch 190 Million Tonne (Mt) level by 2030, according to a report by SteelMint India. The demand will be largely fuelled by construction and infrastructure sectors, which contribute 60-65 per cent to the demand, the market research firm said.

In 2030, India's steel demand is projected to reach 190 Mt based on a 7 per cent Compound Annual Growth Rate (CAGR).

"In the best case scenario, it can also reach 230 Mt by 2030," the report titled 'India's Steel and Coking Coal Demand 2030' stated.

The demand will also be pushed by sectors like auto and engineering, and factors like population growth, growing urbanisation, various government initiatives will be its key drivers.

The demand is expected to touch 120 Mt mark by 2023-end, and production will be at 136 Mt, as per the report.

India's crude steel production is expected to be at 210 Mt by 2030, 45 per cent higher from production levels of 2023.

The Economic Times (26.11.23)

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Chapter Activities

Vijayanagar, Kalpakkam and Chennai, Kolkata, Pune, Hyderabad, Dolvi

Vijayanagar Chapter

1) IIM Vijayanagar Chapter carried out workshop on Bund Stability & Solid Waste Management at JSW Steel Vijayanagar works during 8th - 9th September



2023. The Workshop was inaugurated by Mr. L R Singh, COO Vijayanagar works and Secretary of IIM Vijayanagar Chapter. He highlighted the need of sustainable waste recycling and bund management for steel industries. During the event, presentations were made by the experts from IIT Kanpur, IIT-ISM dhanbad, IIT Roorkee, IIT Guwahati. Workshop emphasised on best methods of preparing the bund, Stabilization and life extension of bunds, Use of modeling tools in bund management and some bund failure case studies. Workshop also deliberated on various hazardous wastes generated in steel industry, its sustainable disposal, recycle or re-use. 2) On 16th October 2023, IIM Vijavanagar Chapter and ISW steel conducted the workshop on "Operation and Maintenance of Reheating Furnaces", at FORUM, Vijayanagar Works. The event was inaugurated by Mr. P K Murugan, President Vijavanagar and Salem works, Mr. L R Singh, COO Vijayanagar works and Secretary of IIM Vijayanagar Chapter, Mr. S C Vishwanath EVP Mills. Mr. P K Murugan in the key note address emphasised on collective approach between all the stake holders for overall development of Indian Industry. Mr. L R Singh highlighted the activities being carried out by IIM Vijayanagar chapter and its importance in building the expertise at ISW. Event had delegates from steel plants (ISW group companies, Tata steel, SAIL, BSL & RSP, AMNS), Water treatment companies (Ion exchange, NALCO), Technology companies (Tenova, Fives), Refractory suppliers (Andritz FBB GmbH, Calderys India, IFGL & Monocon, TRL Korasaki, RHI India, Vesuvius). The event was attended by 60 external and 200 internal delegates.

3) IIM Vijayanagar Chapter at JSW Steel Vijayanagar works, celebrates National Metallurgist Day and organises O P Jindal Memorial Lecture, during the month of November every year. This year National Metallurgist Day celebrations and 3rd O P Jindal Memorial Lecture was organised on 4th Nov, 2023 (Saturday) at Experience centre (R&D auditorium)



• Materials Engineering



in Toranagallu. This lecture series is a tribute to Late Shri Om Prakash Jindal a visionary, successful industrialist, a philanthropist, a politician and a leader.

Mr. L R Singh, Secretary-IIM Vijayanagar Chapter, welcomed the Guests and highlighted the IIM Vijayanagar works activities during the year. During the event the young outstanding practising Metallurgist / Engineers, key contributors to the Chapter and Metals and Materials quiz winners of the Vijayanagar Chapter were felicitated.



This year Prof. N Viswanathan, HOD and Sajjan Jindal Steel Chair Professor, Department of Metallurgical Engineering and Materials Science, IIT Bombay delivered the O P J Memorial Lecture on "Challenges and Opportunities in Indian steel Industry". He highlighted the technical, administrative and human resource issue of the industry and provided the possible solutions to it. He also highlighted the need to work on Blast furnace. He emphasised on building the core competencies in iron and steel making from the undergraduate level. He pushed for immediate need of the industry-institute collaborations. The event was attended by more than 200 members from JSW vijayanagr works and nearby companies such as Hospet steels, Kirloskar ferrous, NMDC etc.

4) IIM Vijayanagar Chapter organised innovation Workshop Day by JSW and Vesuvius on 10th November 2023 at JSW Vijayanagar works to discuss innovations in flow engineering and refractories. The event was organised by Mr. L R Singh, COO Vijayanagar works and Secretary of the IIM Vijayanagar Chapter and Mr. Nitin Jain, Managing Director, Vesuvius India. Both spoke about the importance of refractory management and its role in iron and steel industry. Presentations in the workshop covered reliability improvement through adoption of modern practices, digitalization, automation, and Life enhancement to various refractories used in iron and steel. Workshop focused on new technologies, best practices, innovations and development for better, cleaner, efficient and safer future. Vesuvius presented their latest technologies on Mechatronics solutions, New generation of Mag C bricks, Clean Steel Solutions in Continuous Casting, Technologies to assist in Productivity improvement in Continuous Casting, solution for Pellet DRI & Reheating furnace. Experts from Vesuvius also shared their experiences from various plants around the world and also provided views on issues shared by JSW engineers. The event was attended by 200 delegates from Vesuvius and ISW steel.



Kalpakkam and Chennai Chapters

Dr. Placid Rodriguez Memorial Lecture (PRML) 2023

The 15th Dr. Placid Rodriguez Memorial Lecture (PRML) was organised on 5th October 2023 jointly by Kalpakkam and Chennai Chapters of The Indian Institute of Metals. The memorial lecture was delivered by Dr. Komal Kapoor, Chairman and Chief Executive, Nuclear Fuel Complex (NFC), Hyderabad and Vice President (Non-Ferrous Div.) of IIM on the topic "Role of Texture in Fabrication and Performance of Alloys in Nuclear Applications". The programme was hosted at Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam in hybrid mode. Dr. M. Vasudevan, Chairman IIM Kalpakkam Chapter welcomed the gathering and Dr. Divakar Ramachandran, Director MMG & MSG, presented the genesis of Dr. Placid Rodriguez Memorial lecture,



highlighting the details of the series of lectures conducted since 2009. Dr. B. Venkatraman, Director IGCAR, presided over the event and reminisced the life time contributions of Dr. Placid Rodriguez and complimented the contributions of Dr. Komal Kapoor's to materials research and towards steering NFC to meet the mandates of DAE. Dr. Prasad Reddy, Secretary, IIM Kalpakkam Chapter, introduced the PRML speaker.

Dr. Komal Kapoor made a very comprehensive presentation on the role of processing on texture and its influence on material properties in Zirconium, Iron and Nickel-based alloys of the nuclear industry. The talk covered various technological and scientific aspects of texture in Zr alloys, its measurement, its formation during different processing routes & welding, its effect on delayed hydrogen cracking and design of process flow sheets to control the texture. The speaker also dwelt on detailed texture effects in extruded and annealed 18Cr-ODS tubes as a function of cold work and annealing and elucidated on its microstructural origins. The talk finally touched upon important texture effects in Nickel based alloy 690TT. The memorial lecture was well





Dr. B. Venkatraman, Director, IGCAR presenting the PRML plaque to the invited speaker Dr. Komal Kapoor

attended by over 150 participants with QA session and discussions. The invited speaker was honoured with a memento by Dr. B. Venkatraman, Director IGCAR.

Kolkata Chapter

Activities during October :

1) A membership drive programme was organised on 6th October 2023.

2) A new Student Affiliate Chapter "Kazi Nazrul University Students' Affiliate Chapter" was formed recently under IIM Kolkata Chapter.

3) The EC meeting was held on October 17, 2023 at the office premises of Kolkata Chapter.

Pune Chapter : 16th Dr Dara P Antia Memorial Lecture

The 16th Dr. Dara P Antia memorial lecture was delivered by Prof P. M. Ajayan, currently the Benjamin M. and Mary Greenwood Anderson professor of Engineering at Rice University, on Nov 2, 2023 in the auditorium at College of Engineering, Pune (COEP), Technological University Pune. The theme of the lecture was "Nanoengineered Materials - Challenges and Opportunities." He highlighted the recent advances made in nanotechnology especially in the discovery and development of new nano-materials for a wide variety of applications including their applications in the semiconductors, batteries and as photocatalysts. More than 200 participants attended and many more joined virtually.

Mr. Lalitkumar Pahwa, Chairman, IIMPC welcomed those present on this occasion and briefed them



Dr. Sanak Mishra, former President, IIM presenting Scroll of honor to Prof. Ajayan



on various activities being undertaken by IIM Pune Chapter, Mr. T V Narendran, Chairman, DPA Memorial Lecture Committee and CEO & MD of Tata Steel, former President of IIM, very warmly welcomed Prof. Ajavan, our distinguished speaker, all the distinguished invitees present on this occasion, all the members of IIM fraternity in India and abroad, students and all the delegates who participated in this memorable event. A video film on Dr. Dara Antia was screened as a tribute to his outstanding contributions and the dynamic leadership in advancing the professional interests of the materials science and engineering community in India and abroad. Dr. Debashish Bhattachariee. Vice President, Technology and R&D, Tata Steel introduced the distinguished speaker, Prof. Ajavan. Dr. Sanak Mishra, former President, IIM felicitated. Prof. Ajavan with a memento and a scroll of honor on this occasion.

On behalf of IIM Pune Chapter, Dr. DPA Memorial Lecture Committee and the COEP Technological University Pune (formerly College of Engineering Pune), Prof. N B Dhokey, the Hon. Secretary of the DPA Memorial lecture committee proposed a vote of thanks on this occasion. He expressed his sincere gratitude to Prof. Ajayan and thanked Prof. Sudhir Agashe, Vice Chancellor, COEP Technological University Pune, Mr. TV Narendran, Chairman, DPA Memorial Lecture Committee, esteemed Committee Members and All who associated with this event.



Hyderabad Chapter

A two days short course was organised by IIM Hyderabad Chapter on "Metallurgy for nonmetallurgist-industrial practices" – MNM 2023 on 6th & 7th November 2023 at Hotel Hampshire, Lakdikapul, Hyderabad.

Dr. SK Jha Chairman, IIM Hyderabad Chapter and Shri T Muthukumar, Secretary, IIM Hyderabad Chapter extended a warm welcome to the Chief Guest Shri Jayesh Ranjan, IAS, Principal Secretary, Information Technology, Electronics & Communication (ITE&C) and Department of Industries & Commerce, Government of Telangana. In his address, the chief guest congratulated the Indian Institute of Metals, Hyderabad Chapter for organising the course of industrial importance and stated it is very pertinent in the context of India's initiatives with respect to Atmanirbhar Bharat. In his address, Shri Javesh Rajan, IAS touched upon the role of metallurgy in the context of production of various materials for meeting the defence needs of India. He stated that, these two days course will be useful to both metallurgists and non-metallurgists in upgrading their knowledge and it is an excellent opportunity to interact with various experts in the field.

Dr. Tata Narasinga Rao, Director ARCI, and Dr. R Balamurali Krishnan, Director, Defence Metallurgical Research Laboratory, Hyderabad, several other eminent speakers and members of IIM actively participated in the event.

Various topics that have been covered in the two days' workshop by Eminent speakers from Industries included: Materials' Selection and Design, Melting and Casting, Metal Forming, Welding Metallurgy, Joining Technologies, Powder Metallurgy, Additive Manufacturing, Surface Engineering and Coatings, Heat Treatments, Mechanical Testing, Characterization of Materials, Non-Destructive Testing, Environment degradation and its prevention. The workshop was attended by more than 120 participants across India.

Dolvi Chapter

IIM Dolvi Chapter organised two lectures on 10th November 2023 and 17th November 2023. Both the lectures were delivered by Mr. Neelkant, Manager - R&D in JSW Steel Dolvi works. The topics of the lectures were 'Failure analysis of metallic components in steel plant' and 'Case studies of misinterpretation of laboratory scale test/ investigation data'.

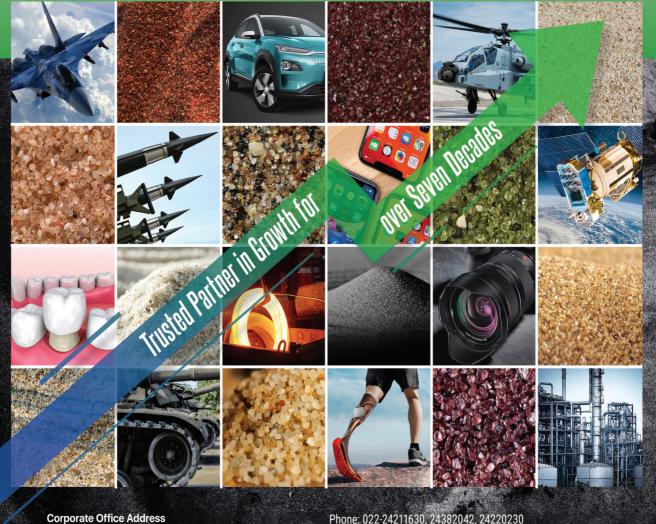


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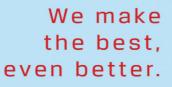
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