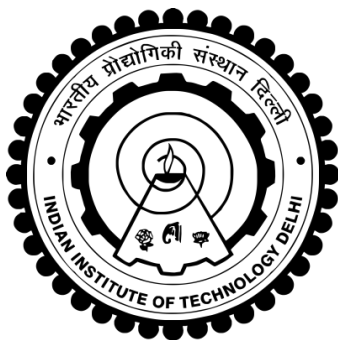


**WELDABILITY AND MECHANICAL CHARACTERIZATION OF A
PRECIPITATION HARDENED CREEP RESISTANT STAINLESS
STEEL**

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**DEPARTMENT OF APPLIED MECHANICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

MAY 2019

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PRECIPITATION HARDENED CREEP RESISTANT STAINLESS
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by

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DEPARTMENT OF APPLIED MECHANICS

Submitted

**in fulfilment of the requirements of the degree of Doctor of Philosophy
to the**



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MAY 2019

Certificate

This is to certify that the thesis, “**WELDABILITY AND MECHANICAL CHARACTERIZATION OF A PRECIPITATION HARDENED CREEP RESISTANT STAINLESS STEEL**” being submitted by Mr. **Dharmendra Kumar Shrivastava** to the Indian Institute of Technology Delhi for the award of the degree of **Doctor of Philosophy** is a bonafide record of original research work carried out by him under our supervision in conformity with rules and regulations of the institute. The result contained in this thesis have not been submitted, in part or in full, to any other University or Institute for the award of any Degree or Diploma.

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Dharmendra Kumar Shrivastava

Abstract

The need to improve the plant efficiency and to reduce fuel & water consumption for lesser per MW infrastructure cost along with the regulatory requirements to reduce SO_x & NO_x, led to the development of technology from Sub-critical to Supercritical and Supercritical to Ultra & Advanced Ultra Supercritical Boilers. The demand for high efficiency in the power plant has resulted in a need to develop materials capable for operating at higher steam pressures and temperatures. In the supercritical boiler, high temperature section of super-heater and re-heater tubes are subjected to the pressure of 24 MPa and temperature of 650°C. The tube materials at this pressure and temperature should be oxidation, corrosion and creep resistant.

The present research work is concerned with the experimental study of recently developed precipitation strengthened creep resistance austenitic stainless steel tube, Super 304H (0.1C-18Cr-9Ni-3Cu-Nb-N; UNS S30432) which is termed as code case 2328-2 by American Society for Mechanical Engineers (ASME). Under the Unified Numbering System (UNS) of ASME, CC2328-2 has been assigned S30432 as its UNS number. To examine the reliability of this material a complete characterization has been done with respect to its mechanical properties and weldability.

Similar and dissimilar welding of CC2328-2 is required for applications in super-heater and re-heaters of supercritical boilers. Welding parameters for similar and dissimilar welding have been established. For welding between austenitic and martensitic stainless steel the available study and literature is by buttering the martensitic steel with Inconel, stress relieving of buttered joint and then welding with austenitic stainless steel to avoid Post Weld Heat Treatment (PWHT). However, this process has its own limitation for mass production. Hence a welding process is developed by direct welding between austenitic (CC2328-2) and martensitic (SA213 T91) steel. This weld has been subjected to post weld heat treatment and there upon microstructure, corrosion resistant property and strength have been studied. Aging and microstructure studies have been carried out to validate the process of welding.

In supercritical boilers, the tubes are given different degrees of bends to maximize heating surface area. The strain developed during bending process, however deteriorates the material properties. For each material, there is a threshold strain value, beyond which if further strain

is given, the material must be normalized or solution treated to regain its properties. No such study for CC2328-2 material has been found. It is observed that ASME has treated CC2328-2 and SA213 347H material as equivalent with respect to strain beyond which solution treatment is required to regain the material properties. However, it is to be noted that both the material differs in chemical compositions and the mechanism of material strengthening is also different. Hence it is felt that a criterion must be developed for CC2328-2 materials by studying the behavior of the material by straining and aging. To understand the precipitation strengthening behavior and effect of strain, CC2328-2 material was strained from 10% to 27% and aged at 650°C for a period ranging from 8 hours to 670 hours and then microstructure was studied using optical microscope. The corrosion resistance property was studied using anodic polarization method & CuSO₄ method.

Precipitation hardened austenitic stainless steels are adversely affected by the precipitation of unwanted brittle phases like sigma & laves phases and are also susceptible to stress corrosion cracking. Study on nature of precipitates after short term aging was carried out by EDX.

सार

बिजली संयंत्र की दक्षता को बढ़ाने की आवश्यकता, ईंधन और जल की खपत प्रति मेगा वाट कम करने का प्रयास और सॉक्स- नॉक्स को कम करने का रेगुलेशन, सभी ने मिलकर सब क्रिटिकल टेक्नोलॉजी से सुपर क्रिटिकल टेक्नोलॉजी और सुपर क्रिटिकल से अल्ट्रा और एडवांस्ड अल्ट्रा सुपर क्रिटिकल टेक्नोलॉजी को जन्म दिया. बिजली संयंत्र की दक्षता को बढ़ाने के निरंतर प्रयास से ऐसे मैटेरियल्स की जरूरत पड़ी जो की उच्च भाप दबाव और उच्च तापमान को सह सके और उनकी गुणवत्ता में कोई कमी नहीं आये. सुपर क्रिटिकल बायलर के सुपर हीटर और री हीटर सेक्शन में बायलर ट्यूब ६५०-degree सेंटीग्रेड के तापमान पर काम करती हैं. इस तापमान पर बायलर ट्यूब्स को ऑक्सीकरण, कोरोजन और क्रीप प्रतिरोधी होना चाहिए ताकि उनकी गुणवत्ता में कोई कमी नहीं आये.

वर्तमान अनुसंधान कार्य, हाल ही में विकसित बायलर ट्यूब्स जो कि प्रेसिपिटेशन स्ट्रेंथन्ड क्रीप रेसिस्टेंट ऑस्टेनितिक स्टेनलेस स्टील की केटेगरी में आता है, के ऊपर निर्धारित है. इसे अमेरिकन सोसायटी फॉर मैकेनिकल इंजीनियर्स (ASME) द्वारा कोड केस 2328-2 के रूप में या सुपर 304H (0.1 c-18Cr-9Ni-3Cu-Nb-N; UNS S30432) के रूप में जाना जाता है । ASME के एकीकृत क्रमांकन प्रणाली (यूनिफाइड नंबरिंग सिस्टम) के अंतर्गत, CC2328-2 को UNS संख्या S30432 दिया गया है । वर्तमान अनुसंधान कार्य इस CC2328-2 ट्यूब की विश्वसनीयता की जांच करने के लिए इसका सम्पूर्ण कैरेक्टराइजेशन के सम्बन्ध में है जिसमे इस मटेरियल की भौतिक गुणवत्ता एवं वेल्डबिलिटी की पूरी समीक्षा की गयी है.

ऑस्टेनितिक और मार्टेन्सिटिक स्टेनलेस स्टील को एक दूसरे के साथ वेल्डिंग द्वारा जोड़ने की प्रक्रिया, जो कि उपलब्ध अध्ययन में है, वह बड़े पैमाने पर उत्पादन में विलम्ब एवं कठिनाई उत्पन्न करती है. इस प्रक्रिया में पहले मार्टेन्सिटिक स्टेनलेस स्टील के उस खंड पे जिसे ऑस्टेनिक स्टेनलेस स्टील के साथ जोड़ना है, Enconel की परत वेल्डिंग के द्वारा चढ़ानी पड़ती है , तत्पश्चात उसे अलग से हीट ट्रीटमेंट करना पड़ता है स्ट्रेस रिलीविंग के लिए और फिर तब उसे ऑस्टेनितिक स्टेनलेस स्टील के साथ वेल्ड कर सकते हैं क्योंकि स्टेनलेस स्टील के पोस्ट वेल्ड हीट ट्रीटमेंट करने से उसकी गुणवत्ता कम हो जाती है.

इस कठिनाई को दूर करने के लिए एक ऐसी पद्धति का प्रयोग वर्तमान अध्ययन में किया गया जिसके द्वारा ऑस्टेनितिक स्टेनलेस स्टील (CC2328-2) और मार्टेन्सिटिक स्टेनलेस स्टील (SA 213 -91) को आपस में सीधा वेल्डिंग के द्वारा जोड़ा गया एक विशेष प्रक्रिया के तहत तथा वेल्डिंग के बाद उस जोड़ का पोस्ट वेल्ड हीट ट्रीटमेंट किया गया. तत्पश्चात इस वेल्डिंग का कोरोजन टेस्ट, माइक्रो स्ट्रक्चर अध्ययन और स्ट्रेंथ टेस्ट किया गया. इस वेल्डिंग जॉइंट पर बायलर के ऑपरेशन के दौरान कैसा प्रभाव पड़ेगा,

इसके जानकारी के लिए बायलर ऑपरेशन के तापमान पर इस बेल्डिंग जॉइंट की एजिंग की गयी, अलग अलग समय के लिए, और माइक्रो स्ट्रक्चर का अध्ययन किया गया यह देखने के लिए कि कहीं इसकी गुणवत्ता कम तो नहीं हो रही.

सुपरक्रिटिकल बॉयलर में हीटिंग सतह क्षेत्र को अधिकतम करने के लिए बायलर ट्यूब्स को विभिन्न डिग्री पर बेंड करना पड़ता है. इस बेल्डिंग के वजह से ट्यूब्स में स्ट्रेन आ जाती है और यह स्ट्रेन मटेरियल की गुणवत्ता में कमी लाती है. सभी मटेरियल की गुणवत्ता इस स्ट्रेन की वजह से प्रभावित होती है. प्रत्येक मटेरियल कुछ हद तक इस स्ट्रेन को बिना गुणवत्ता में कमी लाये सह सकती है. यह हद प्रत्येक मटेरियल के लिए अलग अलग होती है. अगर स्ट्रेन इस हद को पर कर जाती है तो मटेरियल की गुणवत्ता वापस लाने के लिए मटेरियल को नॉर्मलाइजिंग या सलूशन हीट ट्रीटमेंट करना पड़ता है. CC2328-2 ट्यूब मटेरियल की क्या स्ट्रेन की हद है जिससे ज्यादा स्ट्रेन देने पर इसकी गुणवत्ता में कमी आ जाती है और इसकी गुणवत्ता वापस लाने के लिए इसकी हीट ट्रीटमेंट करनी पड़ेगी, इसका कोई ठोस अध्ययन नहीं मिलता है. हालाँकि ASME कोड में इसका विवरण मिलता है पर कोड इसके स्ट्रेन की हद को एक दुसरे मटेरियल SA213 347H की हद को अपनाने के लिए कहती है जो कि तर्क संगत प्रतीत नहीं होता है क्योंकि दोनों मटेरियल के केमिकल कम्पोजीशन और स्ट्रेंथनिंग मैकेनिज्म अलग हैं. अतः यह जरूरी है कि CC2328-2 ट्यूब मटेरियल को विभिन्न डिग्री पर बेंड कर के इसे बायलर ऑपरेटिंग तापमान ६५० डिग्री सेंटीग्रेड पर एजिंग की जाय और इसकी गुणवत्ता का अध्ययन किया जाय. वर्तमान प्रयोग में CC2328-2 ट्यूब मटेरियल को १०% से २७% स्ट्रेन देकर ८ से ६७० घंटे तक ६५० डिग्री सेंटीग्रेड के तापमान पर रखा गया और तत्पश्चात ऑप्टिकल माइक्रोस्कोप के द्वारा माइक्रो स्ट्रक्चर का अध्ययन किया गया तथा कोरोजन टेस्ट एनोडिक पोलराइज़ेशन और कॉपर सलफेट मेथड द्वारा किया गया.

प्रेसिपिटेशन स्ट्रेंथेनेड ऑस्टेनितिक स्टेनलेस स्टील की गुणवत्ता अवांछित प्रेसिपिटेट्स से भी कम हो जाती है, जैसे की sigma & laves फेज़ेज़ अवांछित ब्रिटल फेज़ हैं जो कि स्ट्रेस कोरोजन क्रैकिंग का कारण बन सकती हैं. अतः एजिंग के बाद EDX उपकरण द्वारा इसमें होने वाले प्रेसिपिटेट्स का अध्ययन भी किया गया.

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Nomenclatures and abbreviations

t_r	Time to rupture
h	hour
Ms	Martensite start
TP	Type
G_i	Gibbs initial Energy
G_f	Gibbs final Energy
G_θ	Free Energy per unit volume of θ
r^*	Critical size of radius
Z	Zeldovich Factor
K_B	Boltzmann constant
J	Joule
E _{pp}	Primary Passivation Potential
V	Voltage
P	Pressure
T	Temperature
t	Time
CC	Code case
ASME	American Society for Mechanical Engineers
UNS	Unified Numbering System
ASTM	American Society for Testing of Metals
BCC	Body Centered Cubic

FCC	Face Centered Cubic
MT-DATA	Metallurgical and Thermochemical Databank
SGTE	Scientific Group Thermodata Europe
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
EDX/EDS	Energy Dispersive X-ray Spectrometry
SCC	Stress Corrosion Cracking
IGC	Inter granular Corrosion
KWh	Kilo Watt hour
FG	Fine Grain
LMP	Larsen Miller Parameter
BM	Base Metal
GTAW	Gas Tungsten Arc Welding
PWHT	Post Weld Heat Treatment
AWS	American welding Society
HAZ	Heat Affected Zone
CVN	Charpy V Notch
CLR	Centre Line Radius