

Influence of Microstructure and Second Phase Precipitation by Adding AI-Ti on the Mechanical Behavior of Austenitic Heat Resistant Steel Castings

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Abstract: Heat resistant stainless steel castings are widely used in several industrial sectors working at high temperatures like cement, chemical, heat treatment, metal enameling and other several industries. The purpose of this investigation is to examine the influence of changing microstructures and intermetallic precipitates (Ni₃Al, Ni₃Ti) by heat treatment on the mechanical properties of heat resistant steel castings of type 11Ni-23Cr-0.35C used at temperatures more than 950 °C. Aluminum and Titanium were added to the steel melt before tapping the heat into the casting moulds. The change in microstructures after aging heat treatment (850-900 °C, 85 to 200 h) was detected using scanning as well as optical microscopy. The mechanical properties (high temperature tensile and hardness and creep tests) were measured for all the microstructures obtained in as cast, solution treated and aged conditions. The results showed improved mechanical properties as compared to that for plain heat resistant steel castings, assuring enhancement of life time of treated and aged parts.

Key words: Heat resistant steel castings, solution treatment, gamma prime, chromium carbides, sigma phase, aging, change in microstructure, hot tensile strength, creep strength.

1. Introduction

Heat resistant castings are capable to withstand high temperature operations in excess of 650 °C either continuously or intermittently [1]. They have high carbon content up to 0.7% to improve elevated temperature strength and creep resistance but reduce the ductility [2]. Heat resistant steel casting are widely used in annealing trays, carburizing boxes, radiant tubes, retorts, petroleum still-tube supports and are widely used in cement, chemical, heat treatment and enameling industries [3-5]. The most remarkable alloy is the HH heat resistant austenitic cast steel having 10-12% Ni, 22-25% Cr, 0.3-0.45% C according to

ASTM A608 (HF30) [6].

In this alloy Nickel and Chromium have the greatest influence on improving creep strength, thermal fatigue and oxidation resistance at high temperatures up to 1,000 °C. Nickel (10-12%) and Carbon (up to 0.45%) improve the strength and creep resistance at such high temperature [7, 8]. The presence of up to 2% Si and 2% Mn in this HH alloy contribute to more oxidation resistance and solid solution strengthening [9]. It was reported that long exposure time of that HH alloy at temperatures higher than 850 °C resulted in an offset of its life time due to embrittlement inside and between the dendrite arms of cast structure [9]. In many applications of such alloy in industry, sever adhesive and abrasive forces are induced which increase the propagation of thermal fatigue and accordingly reduce the life time of the alloy [10, 11]. Many researchers tried to improve the

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embrittlement phenomenon and to increase the life time through adding carbide forming elements like tungsten, niobium and vanadium. However, it was reported that addition of niobium to that cast alloy combined some of carbon to form NbC instead of Cr_3C_2 , Cr_7C_3 and $Cr_{23}C_6$, although Nb had affinity to form brittle intermetallic phase with Ni and Si called G-phase [12-15]. However, Al or/and Ti were reported to be beneficial to inhibit the effect of such brittle phase [16].

2. Material Design & Aim of Research

This research aimed to improve high temperature strength, hardness and creep resistance of the HH heat resistant alloy steel. This is only possible by adding aluminum and/or titanium to the alloy during melting with a background that Al forms intermetallic phase with Ni called gamma prime (Υ) phase Ni₃Al which is FCC phase coherent with austenitic base matrix, however, Ti can not only form gamma prime phase (Υ) Ni₃Ti which is coherent with the austenite matrix as well, but also reacts with some of the carbon in the alloy to from TiC that increases the free Cr content, which increases in turn oxidation resistance of the alloy. These Intermetallic precipitations inside the austenite matrix were accomplished by aging the new alloy composition for long time at high temperature (850-900 °C).

3. Experimental Procedure

3.1 Melting and Casting

Four cast-heat resistant steel melts were prepared using clean HH-alloy scrap, aluminum (99.9%), ferrotitanium (48% Ti, 3% Al), ferrochromium (70% Cr, 0.05% C), graphite and nickel pellets. The steel scrap was melted in coreless medium frequency induction furnace. After complete melting addition of CaO-alumina slag to the top of the molten steel in order to protect it from further oxidation and to absorb any impurities coming up from the melt during induction stirring. Additions of Al metal, FeTi, Graphitic Carbon, FeCr and Ni pellets were only done after the first sample analysis to correct the final composition of the alloys under investigation. Pure plain carbon steel scrap can be used to correct any increase in the alloy contents. The casting temperature was adjusted at 1,580 °C using dipping thermocouple. Every melt was tapped and cast into preheated, ZrO₂ sprayed, Y-block cavity according to DIN EN 1536 type 3. The lining of the induction furnace was chosen to be high alumina lining.

The chemical composition of the prepared Y-block castings is shown in Table 1.

3.2 Heat Treatment

The solidified Y-blocks together with their risers and in-gates were homogenized annealing at 950 °C for three hours to remove segregation and relief stresses generated during solidification. After air cooling, the castings were blasted with steel shots and then all its in-gating system was cut. The Y-blocks were shot blasted second time to remove new scale and processing spatter.

Solution treatment of the Y-blocks were done at 1,000 °C for 2 h and then water quenching (WQ) to re-dissolve carbon into austenite and remove any precipitation of both sigma (σ) and G-phases. Test samples for microstructure and hardness, tensile, creep were machined from the Y-blocks. Final aging heat treatment of the samples was achieved at 850-900 °C for 85 to 200 h.

 Table 1
 Chemical composition of the experimental modified HH-Casts.

Allow	C	C;	Mn	Cr	Ni	Mo	A 1	Ti	
Alloy	U	51	IVIII	U	INI	IVIO	Al	11	
HH	0.35	1.56	1.35	24.62	11.36	0.32	0.03	0.01	
HH-1	0.37	1.48	1.41	24.31	12.03	0.23	0.92	0.00	
HH-2	0.34	1.53	1.39	23.94	11.85	0.21	0.81	0.25	
HH-3	0.32	1.46	1.38	24.74	12.04	0.15	0.79	0.47	

4. Results and Discussion

4.1 Changes in Microstructures after Aging

The microstructures of the cast alloys (HH, HH1, HH2, and HH3) are nearly the same in as cast conditions as shown in Fig. 1, where the austenitic phase is surrounded by narrow fingers and/or islands of ferrite. Dispersions of chromium carbides, cementite (Fe₃C) and TiC can be observed at grain boundaries or inside the dendrite arms. Ferrite islands are formed inside grain boundaries and surrounded with Cr_7C_3 , $Cr_{23}C_6$, TiC and sigma (σ) as well as G-phases.

It is evident that the as-cast structure has coarse austenite grains (ASTM No.3) with wide boundaries, and the dendrite arms co-inside along with the grain boundaries. Massive precipitations of chromium carbides inside the grain boundaries are observed, however fine TiC are distributed inside austenite and its grain boundaries.

Solution treatment of the cast samples resulted in grain refinement and narrowing the grain boundaries as shown in Fig. 2, due to the partial dissolution of carbon in austenite and the minimum time for grain growth. It can be observed that the transformed ferrite phase is still there inside or near the grain boundaries of austenite; however, some of TiC and the rest of Cr_7C_3 , $Cr_{23}C_6$ are coagulated inside grain boundaries.

The changes in microstructures occurring after aging the solution treated cast samples (850-900 °C, 85-200 h, air cool) can be observed as in Fig. 3, where fine intermetallic compounds of Ni, Al and Ti are precipitated inside the austenite phase. The density and grain size of the precipitated hard particles of (Υ) phase depended on the time span of aging process; however, coagulation of gamma-prime phase took place



Fig. 1 As cast microstructures of HH, HH-3 heat resistant steel casts.



Fig. 2 Microstructures of solution treated heat resistant casts, (1,100 °C, 2 h, WQ).

at 200 h aging time for the alloy HH-1 having 0.92% Al. Addition of Ti to that alloy refined the gamma-prime phase due to the presence of TiC which pin the matrix preventing further grain growth or coagulation of the second phase.

4.2 Mechanical Testing

4.2.1 Hardness

Hardness of the heat treated investigated cast alloys were measured at room temperature using IDENTIC universal measuring machine and an average of three tests are reported in Table 2. The results show that the hardness of as cast alloys (110-121 HB) are increased by aging depending on the aging time at specific temperature. Solution treatment resulted in only slight increase in hardness for all alloys due to the resolution of some carbide into the austenite which increases the strain hardening capacity of austenite. The maximum hardness is obtained for alloys HH-1, HH-2 and HH-3 after 200 h reaching 265, 300, and 400 HB respectively.

Fig. 4 illustrates the trend of hardness measurements for all alloys aged at 900 °C for 200 h. It is clear that aging of cast alloy at any temperature and time resulted in only slight increase in hardness while the presence of Al and Ti in other alloys contributes to higher hardness due to the formation of intermetallic compounds as mentioned earlier.

4.2.2 Tensile Strength

Fig. 5 shows the trend of tensile strength measured at room temperature for all alloys after aging at 900 °C and 200 h. Alloying with 0.9% Al increased the strength

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Alloy	As-Cast	Solution treated	Aged 890 °C 85 h	Aged 900 °C 100 h	Aged 850 °C 150 h	Aged 900 °C 200 h
HH	110	116	116	117	115	200
HH-1	115	119	121	123	220	265
HH-2	116	121	132	130	239	300
НН-3	121	124	137	139	275	400

 Table 2
 Hardness of produced, heat treated cast heat resistant steels.



Fig. 4 Hardness of the produced alloys in as-aged conditions (900 °C, 200 h).



Fig. 5 Tensile strength of aged alloys (900 °C, 200 h).

to about 300 MPa, while addition of about 0.47% Ti increased the strength to about 458 MPa for alloy HH-3. This increase in strength is nearly double that for cast alloy HH. The precipitation of Ni-Al-Ti gamma prime phases hardens the austenite matrix for that maximum strength; however, the strength of alloy HH-1 is increased moderately after aging for 200 h due to the coalescence of precipitates together producing blocky non-homogeneous precipitates.

4.2.3 Creep Strength

The aged cast heat resistant alloys were subjected for long time creep tests at a stress of about 115 MPa and a fixed temperature of about 800 °C. The creep behavior of those alloys are projected as in Fig. 6, which illustrate that the HH alloy was failed after about 1,000 h after strained by about 8%, however alloy HH-3 resisted till a life of about 8,000 h after strained by about 12%. Alloys HH-1 and HH-2 failed Influence of Microstructure and Second Phase Precipitation by Adding Al-Ti on the Mechanical Behavior of Austenitic Heat Resistant Steel Castings



Fig. 6 Creep behavior of the produced cast alloys HH, HH-1 to HH-3.



Fig. 7 Fracture surfaces for alloys crept at 800 and 115 MPa. after 4,300 h and 6,000 h at strain values 15% and 23% respectively. It is evident that the hard strong

matrix of alloy HH-3 has long life time but stiff to be strained and failed after 12% as compared with both

HH-1 and HH-2 alloys that strained till 15% and 23%. Accordingly, the alloy designer has to choose HH-2 or HH-3 alloy according to the applied forces at 800 °C. The intermatallic compounds formed during aging of alloys HH-2 and HH-3 strengthen and harden the austenite matrix preventing it to fail after short loading time, in other words alloying of HH cast steel with both Ti (0.25-0.47%) and Al (0.81-0.79%) together gives the maximum performance of such alloy even over than that for alloy containing 0.9% Al. The appearance of the crept fracture surfaces are depicted in Fig. 7, where at least 80% ductile fracture is observed for alloy HH-2, however, some brittle facets can be observed in fractured alloys HH and HH-3. It can be observed also that the separation of grains during fracture at high temperature is enhanced at the peripheries of Ni₃Al and Ni₃AlTi intermetallic Y precipitates as shown for alloys HH-2, and HH-3 in Fig. 7.

5. Conclusions

From the above mentioned measurements and discussions it can be concluded that:

Additions of Al (0.79-0.92%) and /or Ti (0.25-0.47%) to HH-base alloy form with Ni intermetallic precipitates in the austenite matrix after aging heat treatment (850-900 °C, 85-200 h).

It was proved that the hardness and strength of the developed HH alloy were increased to reach 400 HB, 458 MPa respectively using Al-Ti combined alloying,

The creep resistance of the developed HH alloy reached 6,000 to 8,000 h at strains 23 and 12% when alloyed with Al and Ti. The fracture appearance after creep proved to be highly ductile as compared to that for HH-base alloy.

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References

- ASM Handbook. 2005. "Casting of HR-Alloys." ASM International, Materials Park, Ohio, Vol. 15.
- [2] Read, C. 2006. *The Superalloys, Fundamentals & Application*. Cambridge, UK.
- [3] ASM Handbook. 2005. "Properties and Selection Iron, Steel, and High Performance Alloys." ASM International, Materials Park, Ohio, Vol. 1.
- [4] Donachie, S. J. 2002. Superalloys A Technical Guide Second Addition. ASM Publication.
- [5] Maziaz, P. J. and Evans, N. D. 2010. "High-Temperature Mechanical Properties and Microstructure of Cast Ni-Based." In *Proceeding of EPRI Conference*, Santa Fe, USA.
- [6] Schafrik, R. and Sprague, R. 2004. "Saga of Gas Turbine Materials: Part III." Advanced Materials and Processes 162: 27-30.
- [7] Tanaka, R. 2000. "Research and Development of Ultra-High Temperature Materials in Japan." *Materials at High Temperatures* 17 (4): 457-64.
- [8] McLean, M. and Dyson, B. 2000. "Modeling the Effects of Damage and Microstructural Evolution on the Creep Behavior of Engineering Alloys." *Journal of Engineering Materials and Technology* 122: 273-8.
- [9] Rae, C. and Cox, D. 2000. "On the Primary Creep of CMSX-4 Superalloy Single Crystals." *Metallurgical and Materials Transactions A* 31A (9): 2219-28.
- [10] Dablonski, P. and Maziasz, P. J 2012. "Processing of Advanced Cast Alloys for A-USC Steam Turbine Applications." J. Miner. Metals Mater. Soc. 64: 271-9.
- [11] Zeytin, H. K. and Tekín, A. 2006. "Microstructural Evolution in a Ni-Cr-Co Based Superalloy during Cooling from the Melt." *Mater. Charact.* 57: 86-93.
- [12] Long, F. and Jeong, H. W. 2009. "Phase Transformation of and σ Phases in an Experimental Nickel-Based Superalloy" J. Alloy. Compd. 478: 181-7.
- [13] Piekarski, B. 2001. "Effect of Nb and Ti Additions on Microstructure of Ni-Cr Cast Steels." *Mater. Charact.* 47: 181-6.
- [14] Karaminejad, M. 2005. "The Fracture Mechanism of an Austenitic Cast Heat Resistant Steel." Int. J. ISSI 2: 31-6.
- [15] Haitao, W. and Gaunghui, M. 2009. "Effect of Al and Si on High Temperature Oxidation of Cast Heat Resistant Steel." *Trans. Tianjin Univ.* 15: 457-62.
- [16] Wu, X. and Zhi, X. 2007. "Effect of Ti on the Morphology Morphology of Carbides." *Mater. Sci. Eng.* A. 457: 180-5.