OPTIMIZING MICROSTRUCTURE AND MECHANICAL PROPERTIES OF CAST AISI-H11 HOT WORK TOOL STEEL BY ALLOYING WITH NIOBIUM AND BORON

W. ELGHAZALY¹ & R. RASHAD²

¹Researcher at Central Metallurgical R&D Institute, Egypt, Helwan

²Professor at Mechanical and Production Engineering Department, Cairo University, Egypt

ABSTRACT

Hot work tool steel like grade AISI-H11 is used extensively to manufacture dies for metal forming like die casting, forging and extrusion dies due to its good toughness to hardness ratio and good resistance to offset fatigue strength. However, there are many application drawbacks like prior austenite grain gross and surface cracking of dies after short service life time. New cast H11 steel containing niobium and boron as alloying elements has been designed in a trial for better structure stability, higher toughness and fatigue resistance as well as low production cost. The mechanism is to increase strength and toughness through grain refinement of the steel microstructure even after many thermal service cycles through alloying with such elements. Experimental cast hot work tool steel dies with different alloy additions were produced, machined, heat treated and then mounted into Al-Si die casting machine to evaluate the changes in microstructure and mechanical properties related to its thermal life cycles. Universal testing machines to detect hardness, Charpy V-notch impact toughness and bending fatigue together with metallurgical microscopy, image analysis microscopy, SEM=EDX and X-ray diffraction techniques were used to measure the mechanical and microstructure properties of the produced steel alloys. The results showed improvement in microstructure grain refinement and mechanical properties of H11 cast hot work steel dies when alloying it's melt with 0.3% B + 0.4% Nb. Improved hardness (48 HRC) and toughness (29.5 J) together with more bending fatigue life time to reach one million (1 M) cycles when applying mean stress at 750 MPa were obtained for such cast modified steel alloy.

KEYWORDS: High Pressure Die Casting Dies, Hot Working Dies, Microstructure, Grain Refinement, Hardening, Tempering, Mechanical Properties, Fatigue Strength & Impact Toughness

Received: Apr 18, 2020; **Accepted:** May 08, 2020; **Published:** Jul 13, 2020; **Paper Id.:** IJMPERDJUN2020337

INTRODUCTION

Hot work tool steel grade AISI-H11 is widely used for various applications such as dies for hot forging, dies for high pressure die casting (HPDC) of aluminium, magnesium, copper and their alloys, and forming moulds like in glass industry. etc. 1, 2 The mechanical forces acting on dies used in Aluminium pressure die casting under temperature range 650-750°C reported to cause many faults after specific working cycles due to wear, cracking, subsurface damage and changes in geometry of the dies. 3, 4, 5 On the other hand dies always experience high cyclic temperature changes, fluctuating mechanical loading, erosive-corrosive processes during operation and consequently suffer loss of fatigue strength. 6, 7, 8

Implementing such steel grade in high temperature pressure die casting sometimes is not satisfactory when exposed to severe friction or direct thermal contact with molten metal during service for long span of time due to unexpected grain gross of prior austenite and loss of toughness. 9 It was reported that stabilizing and pinning the microstructure of alloyed steels at different temperatures depends to a great extent on the grain refinement of prior

austenite grains before heat treatment. 10,11Additions of alloying elements like Niobium, boron and Titanium act as carbide and nitride hard phases that creating obstacles preventing further grain growth specially after cyclic heating and cooling operations as in metal die casting. 12, 13

In this research a trial was made to produce and implement pressure casting dies with increased toughness, hardness and fatigue strength, over that experienced using the standard AISI-H11 hot work tool steel dies, by adding alloying elements such as Boron (0.11-0.31%), Niobium (0.15-0.40%) to molten H11 alloy bearing Nitrogen (0.07%), in order to grain refine and strengthen the matrix after solidification and heat treatment. Changes in microstructure, hardness, toughness and bending fatigue strength are measured for all steel compositions and are compared to those obtained from the standard H11 hot work steel dies.

EXPERIMENTAL PROCEDURE

Material Preparation

In this research six cast alloys of standard H11 and modified steels marked H11, Cast1 to Cast5 were melted in induction furnace lined with alumina using clean scrap of decommissioned H11 dies and also scrapped ones created during machining or faulty heat treatment. After complete melting the slag is skimmed away and new slag composed of 60% CaO -30% Al₂O₃-10% CaF₂ were added in order to enhance absorbing any impurities and protect the metal surface from further oxidation by atmospheric oxygen. Final analysis corrections of main molten steel alloy contents were done, then nitrogenated –FeMn were added deep into the molten steel to increase its nitrogen content to about (0.07% N) in average and finally variable quantities of FeNb and FeB ferroalloys were added to every heat patch five minutes before pouring. The nitrogen is intended to be added during melting of such steels and before adding secondary carbide and nitride formers (Nb and B) to take advantages of its well-known strengthening effect of tool steels and to ensure formation of BN. The average chemical composition of the produced alloys as analysed using spectral analysis is shown in Table 1.

Table 1: Chemical composition of produced cast alloys

Steel alloy	C wt. %	Si wt. %	Mn wt. %	Cr wt. %	Mo wt. %	V wt. %	B wt. %	Nb wt. %	Al wt. %	S wt. %	N wt. %
H11	0.37	0.95	0.63	5.23	1.49	0.47	0.00	0.00	0.03	0.02	0.08
Cast1	0.38	0.89	0.59	5.07	1.50	0.51	0.11	0.15	0.02	0.01	0.06
Cast2	0.35	0.90	0.70	4.92	1.51	0.49	0.22	0.22	0.03	0.02	0.06
Cast3	0.36	0.92	0.61	5.03	1.49	0.46	0.00	0.29	0.02	0.01	0.07
Cast4	0.37	0.90	0.72	5.14	1.38	0.50	0.31	0.40	0.03	0.01	0.08
Cast5	0.38	0.93	0.59	4.95	1.31	0.46	0.00	0.35	0.02	0.03	0.08

The molten metal was cast in preheated (400°C) chemically bonded sand mould designed to produce sound steel plates (400x600x50 mm) ready to be machined and heat treated for the use in pressure die casting of eutectic Al-Si alloy at Universal company for house ware industries as shown in Figure 1. Electrical discharge machining (EDM) technique was chosen to precise machining the steel plates and forming the cavities, ingates and sprues in order to ensure high quality produced castings as shown in Figure 2. On the same time Charpy – V notch, fatigue samples and coupons for microstructure examinations were machined by the same technique (wire cut and spark erosion) from all cast plates before and after usage in pressure die casting machines.

Figure 1: Casting dies mounted on the holders opened to remove Al-Si- cast parts.

Figure 2: Typical produced Al-Si castings for gas stoves.

Heat Treatment Cycles

The test specimens were heat treated according to the chosen cycles; meanwhile the optimum heat treatment conditions were then applied to the produced experimental steel dies before assembling them into the die casting machines. In the first cycle all the steel samples were annealed in muffle furnace at 1000 °C for 2 h and then furnace slowly cooled in order to soften the structure (pearlite+bainite+carbides) and remove casting metals segregation and stresses. In the second cycle all the annealed samples were grain refined by normalizing at 850°C for one hour and then still air cooled, to grain refine the coarse structures and getting more homogenization. Hardening of all test samples was done by quenching in oil after stepwise heating to reach austenitizing temperature of 975°C, hold for one hour (975°C/1h-oil quenching) and then tempering was done at different temperatures 400 to 700°C to assess its influence on toughness and hardness. The optimum tempering temperature range that ensured good combination of toughness and hardness for all the tested samples was then chosen to temper the produced plates with optimised composition. In all cases the test samples and the produced cast plates are double tempered in the vicinity of the optimum tempering temperature (550-580°C/2h-still air cooling).

The rate of heating and sequence of heat treatment for both hardening and tempering is projected as in Figure 3. Stepwise heating used in hardening was fully followed in normalizing of all samples and the produced plates.¹⁵

Surface hardening of high pressure die casting dies is reported to be beneficial in increasing their adhesion and abrasion resistance as well as their endurance limit.¹⁶ In this investigation it is not intended to surface treat the produced cast alloys after the through thickness or full heat treatment as described above but may be investigated in further investigation by introducing new ideas and technologies.

Figure 3: Heat treatment cycles used for hardening and tempering cast steels

Testing of Microstructures and Mechanical Properties

The microstructures of all produced alloys are investigated in as-cast, quenched and double tempered conditions using inverted laboratory Olympus type-light microscopy, while assessment their phases qualitatively and quantitatively are done using JEOL-JSM-7600F type scanning electron microscopy (SEM) aided with emission diffraction X-ray (EDX) in situ analysing probe. The percentages of microstructure phases like martensite, bainite, retained austenite, carbides and nitrides are measured using X-ray diffraction unit (phase intensity I, versus 2θ degrees). Image analysing microscopy was used to measure the ductile to brittle fracture ratio and helped with the aid of ASTM-grain size charts in assessing the degree of grain refinement of the produced steel microstructures.

The values of mechanical properties like hardness (HRC) and toughness (Joules) are obtained by testing samples using IDENTIC-hardness testing machine and Charpy-V notch pendulum testing machine respectively. Bending fatigue strength is measured for all steels under investigation using R.R. Moore type rotating bending fatigue test machine with standard cylindrical samples machined according to ASTM-E466 type specimen having 9.35mm main diameter, 6.35mm thinnest (breaking point) middle diameter and length of 100mm. The fatigue test samples were thoroughly cleaned after heat treatment using fine blast shooting and then polished to obtain clean smooth surface.

RESULTS AND DISCUSSIONS

Analysis of Microstructure Changes

The changes in microstructure of the produced cast steels in as-cast and after heat treatment conditions were investigated using both optical and scanning electron microscopes bearing in mind the cooling rates and phase transformations predicted in the standard continuous cooling transformation (CCT) diagram of AISI H11 steel which can be applied to the alloys produced with minor changes as shown in Figure 4, 17. It is clear from the diagram that the rate of cooling from austenitizing temperature influences to a great extent the final microstructure characteristics and hardness of the steel.

Figure 4: Continuous Cooling Transformation diagram of H11 hot work tool steel 17

During normalizing heat treatment cycle (850°C-1.5h) and after slow air cooling rates, the microstructures of the produced steels were changed rapidly to ferrite-carbide aggregates and some pearlite phases which in turn resulted in grain refinement of the matrix and relief disorders and segregations. Figure 5 illustrates the changes in normalized microstructures, where considerable grain refinement of the matrix was obtained for the Cast4 alloy (grain size No. 6 as in ASTM E112 charts) if compared with those for the Cast H11, Cast2, Cast3 and Cast5 alloys (grain size No. 2-4).

Normalized Cast-H11 Normalized Cast5 (H11+0.35% Nb)

Normalized Cast4 (H11+0.3%B+0.4%Nb)As Cast-H11

Figure 5: Grain refinement of normalised cast microstructures [850°C-1.5h-AC]for the Cast H11, Cast3 and Cast4 alloys.

Final hardening of the cast steel plates and their samples by quenching from austenitizing temperature (975°C/1h-oil quenching) altered the microstructures to martensite-retained austenite-different carbides as shown in Figure 6-A. However and to ensure tough strong matrix of cast steels, double tempering was done at 550°C and then 580°C for 2h, (580°C/2h-still air cooling) in order to rearrange dislocations and remove quench stresses. In the same time and during the first tempering process retained austenite transformed to martensite, while after the second tempering stress relief and reorientation of dislocations in the new transformed martensite took place. Figure 6-B depicted the double tempered microstructure of steel Cas4 alloy which is composed of tempered martensite, primary carbides (especially Cr₇C₃) and secondary carbides (NbC) and nitrides (BN) precipitations that ensured final hard, tough and strong steel matrix.

The mechanism of hardening in such steel alloy is that during austenitizing at high temperature all Fe-Cr carbides were dissolved into austenite and then precipitated again from 890 °C during quench cooling and the presence of NbC and BN secondary precipitations induced hard obstacles hindering any grain growth of prior austenite grains and forced martensite to be transformed within such small grains which in turn produced grain refined, hard and tough matrices. It is well established in other researches that there are relations correlating the grain size of steel phases and its yield strength, cleavage fracture stress and the ductile to brittle transition temperature as follow; 18-19

- 1-The classic Hall-Petch equation relates the yield strength (σ_y) to the grain size (d):
- $\sigma_y = \sigma_0 + K_y d^{-1/2}$
- Where: K_y the Hall-Petch slope and σ_0 is constant.
- 2- Similar equation applies to cleavage fracture stress (σ_f) and grain size (d):
- $\sigma_f = K_f d^{-1/2}$
- 3- Similar equation applies to ductile to brittle transformation temperature (TB):
- $TB = T_0 - K_B d^{-1/2}$
- 4-The fatigue strength is often taken to vary with grain size in the same way of Hall-Petch equation.

Accordingly, the mechanism of grain refinement practically not only upgrades the yield strength, hardness, toughness and fatigue strength but increases the resistance to brittle cleavage of the steel alloys.

A- As quenched Cast4B- Double tempered Cast4

Figure 6: Microstructures of as quenched and double tempered of the Cast4 alloy

Figure 7: X-ray diffraction count intensity (I) for martensite, carbides and nitrides in the double tempered Cast4 alloy.

The characteristics of the optimized microstructure of Cast4 (alloyed with 0.3% B+0.4% Nb) after hardening and double tempering heat treatment process are depicted as in Figure 7, which projects the intensity (I-count) and types of phases as measured using X-ray diffraction. This structure which contains 70% martensite, 21% primary carbides and 9% secondary carbides and nitrides is considered the optimum grain refined, homogenous and hard texture for that alloy composition Cast4 over other steels alloyed with lower contents of Niobium and Boron.

Mechanical Properties

The heat treated and tempered test samples were subjected to testing for hardness, toughness and bending fatigue according to the ASTM standards mentioned earlier, every predicted test result is indeed the average of at least 3 to 5 tests and the

deviation of the all tests did not exceed $\pm 2\%$.

Toughness

The major properties that can influence the life time of hot work steel die are toughness and hardness, Toughness helps steel to withstand sudden thermal and repeated shocks, while hard matrix increases its wear resistance at high temperature and hence increased resistance to crack initiation. Charpy V-notch (CVN) and hardness Rockwell C (HRC) were measured for all heat treated and double tempered samples according to ASTM standards. The trend of toughness measurements as function of final tempering temperature is shown in Figure 8, where steels produced like the Cast4 and the Cast2 alloys have the highest toughness of about 29.5J and 27.9J after tempering at (580°C/2h-AC) respectively if compared with that for the Cast H11 alloy (20J), the Cast3 alloy (22.5J) and the Cast5 alloy (24J). The addition of niobium (0.22-0.4%) and boron (0.2-0.3%) to the Cast H11 alloy revealed higher toughness values if compared to that for steels alloyed with niobium addition only or the standard Cast H11 steels, due to their strong grain refining of prior austenite phase, strengthen of matrix by interstitial solid solution and pinning the matrix against further grain growth during service by their stable NbC and BN precipitates.

Figure 8: Toughness (CVN) values measured versus tempering temperature.

Hardness

Hardness of the investigated steel alloys was measured using hardness Rockwell C test according to ASTM E18. The hardness values for all steels decreased as the tempering temperature increased as projected in Figure 9. The steel alloy Cast4 shows an optimum hardness value (48 HRC) after double tempered at 550 –580°C for 2h and then still air cooled, this can be attributed to the induced grain refining effect in the steel matrix created by precipitation of secondary carbides and nitrides of niobium and boron, while the rolled heat treated Cast H11 alloy gave only 45HRC after applying same heat treatment cycles.20.

Figure 9: Hardness values measured versus tempering temperature

Fatigue Strength

Bending fatigue strength is measured for Cast4-which composition showed maximum toughness and hardness values over the other cast steel produced - using standard cylindrical samples machined according to ASTM-E466 type specimen having 9.35mm main diameter, 6.35mm thinnest (breaking point) middle diameter and length of 100mm. The fatigue test samples were thoroughly cleaned after heat treatment using fine blast shooting and then polished to obtain clean smooth surface. Figure 10 shows the results of bending fatigue tests as a relation between the operating stresses and the number of cycles (S-N) till fracture.

It is evident that cast steel alloyed with 0.3% boron and 0.4% niobium (Cast4 alloy) showed better fatigue strength than the Cast H11 and Cast 5 alloy which alloyed only with Nb. The fatigue strength of the steel Cast4 alloy reached 700-750MPa to perform one million (1M) cycles; however the steelCast1 alloy and Cast5 alloy reached at just 0.75 million (0.75 M) cyclesat applied stress of about 580-600MPa and 680-700MPa respectively, meanwhile the wrought heat treated H11 hot work same dies performed just one million (1 M) cycles at mean applied stress of about 700MPa.

The increase of performance at that fatigue stresses for the steel Cast4 alloy is attributed to the dual mechanism created to strengthen the matrix after heat treatment hence the alloying with 0.3% B+0.4% Nb induced good grain refinement, creating hard carbides and nitrides that pinned any further grain growth at high temperature and the interstitial solid solution hardening action created by boron, all increased the matrix toughness and strength which in turn contribute to optimized fatigue strength and the whole performance of the die.

Figure 10: Bending fatigue plots for the heat-treated H11 steel and the Cast1, Cast4 and Cast5 alloys

Fracture Appearance

The surfaces of the fractured dies or fractured impact samples of steel Cast 4 alloy were prepared for investigation using SEM-EDX unit and the results showed the presence of ductile facets together with cleavage areas as projected in Figure 11. The composition of secondary carbides and nitrides that appeared inside the fracture facets advocated the strengthening mechanism created by them in the vicinity of ductile areas, however the dominating primary chromium carbides especially Cr_{23}C_6 and Cr_7C_3 carbides concentrated in the cleavage brittle areas.

The appearance of fracture surface and the performance of the produced steel Cast4 alloy showed that the modified Cast steel high pressure die casting dies alloyed with 0.3%B and 0.4%Nb proved to compete the same dies manufactured from wrought hot work H11 steel at the same working conditions. This would save tremendous cost and time in manufacturing such high pressure die casting dies through cast tooling technologies rather than the long expensive route of traditional rolling and forging techniques.

Figure 11: Typical fracture surface of the Cast4 alloy composed of ductile facets (42%), cleavage areas, secondary carbides (NbC) + nitrides (BN) and primary Cr carbides precipitates.

CONCLUSIONS

*Significant grain refinement of the normalized Cast H11 die steel was obtained by adding 0.3%B and 0.4%Nb to its nitrogen bearing (0.07% N) chemical composition. Double tempering of that alloy resulted in optimum microstructure which is composed of tempered martensite, well distributed primary chromium carbides, secondary phases of niobium carbides and boron nitrides.

*The optimized composition of cast steel (Cast4) alloy showed maximum hardness (48 HRC) and toughness (29.5J) after quenching and double tempering at 550 °C and 580°C for 2h and then still air cooled.

* Bending fatigue strength limit of the modified cast steel (Cast4) alloy offsets after one million (1M) cycles when applying mean bending stress at 750MPa; meanwhile the wrought heat treated H11 hot work same dies performed just one million cycles at mean stress of about 700MPa.

* The results showed that alloying H11 cast steel melts bearing nitrogen with 0.3%B and 0.4%Nb proved to increase the performance life time of the dies at more applied stress if compared to the same dies manufactured from wrought hot work H11 steel at the same working conditions. This would save tremendous cost and time in manufacturing such high pressure die casting dies.

ACKNOWLEDGEMENTS

Authors would like to thank Universal Company for stoves and engineering industries, Cairo University and Modern foundries – Egypt for giving the facilities to produce and apply the precise high pressure casting dies. Thanks are also for the many colleagues in the Central Metallurgical R&D Institute for giving help in evaluating microstructures. Sincere thanks are also for the Arabic Organisation for Industrialisation (AOI) -Egypt for help to measure fatigue strength of all samples.

REFERENCES

1. S. Naimi, M. Hosseini "Tool steels in die casting utilization and increased mould life" *Research Gate, Advances in mechanical engineering*, Vol.7, issue 1, 2 (2015)
2. O. Barrau et al. "Analysis of the friction and wear behaviour of hot work tool steel for forging" *Wear*, 255, 1444-1454. (2003)

3. A. Ayabeet. Al. "effect of High Thermal Conductivity Die Steel in Aluminium Casting"
4. *International Journal of Metal Casting-Springer* 08, p.47 (2008)
5. W.E. Bryson, "Heat Treatment, Selection, and Application of Tool Steels" 2nd edition, HanserGardner Publications, Cincinnati (2005).
6. D. Wang, I. Schruoff, E. Meurisse "Modern tool steels for long life time in hot stamping Applications" research Gate, Conf. Paper, DOI: 10.1142/9789813207301-0062, 3 (2017)
8. 6. L. Sabine, R. Farhadi "Topological and metric properties of microscopic crack patterns"
9. *Journal of Physics D: Applied Physics*, 46, 29, 295-301.(2013)
10. 7. C. Guy, W. Martin "Fine blanking tool steels" Elsevier, *Procedia engineering*, 183, p.45-52(2017)
11. 8. A. Gunnarsson, B. Hogman "The Influence of steel grade on tool life in hardened tool steels" Sixth Int. tooling conf., p. 1200-1215, Uddeholm Sweden (2011)
12. 9. A. Persson et. al. "Simulation and evaluation of thermal fatigue cracking of hot work tool steels" *International Journal of Fatigue*, 26, 1095-1107. (2004)
13. 10. Y. Yashi, et al, Tempering stability of Fe-Cr-Mo-W-V hot forging die steels, *International Journal of Minerals Metallurgy and Materials*, 24, 1145-1157, (2017)
15. 11. M. Lin, et al, Microstructure Evolution and Carbide Precipitation in a Heat-Treated H13 HotWork Mould Steel. *Metallography, Microstructure and Analysis, Symposium, Tokyo, 2016*
16. 12. L. Jon, T.Dossett "Heat treating of irons and steels" ASM handbook, Vol.40, (2014)
17. 13. M. Fares, et al, "Effects of Conventional Heat Treatments on Mechanical Characteristics of New Hot Working Tool Steel" *Material Science Engineering*, 28, 012-042. (2012)
18. 14. G. Pusb, et al, "Heat Treatment Optimization and Properties Correlation for H11-Type Hot-Work Tool Steel" *Metallurgical and Materials Transactions A*, 49, 455-462, (2018)
19. 15. I. Souki, P. Lours, "Influence of heat treatment on the fracture toughness and crack Propagation in 5% Cr martensitic steel" *Procedia Engineering*, 10, 631 – 637. (2011)
21. 16. I. McAdams "Improving Shot Sleeve Life Through selected Surface treatment" *International Journal of Metal Casting*, Volume 10, Issue 1, DOI 10.1007/s40962-015-0012-4, (2016)
22. 17. G. Roberts, G. Krauss, *Tool Steels*, 5th edition, American Society for Metals, Metals Park, Ohio (1998)
24. 18-P.Lehto et. al."Influence of grain size distribution on the Hall-Petch relationship of welded steels" *Materials science and engineering A*, vol.592, issue 1, p.28-39 (2014)
25. 19-S. Matsuda et.al."Toughness and effective grain size in heat treated steels" *Transactions of iron and steel institute of Japan*, vol.12, issue 5, p.325-333 (1972)
26. 20. Y. Guanghua, et al, "Effects of Heat Treatment on Mechanical Properties of H13 Steel" *Metal Science and Heat Treatment*, 52, 7–8, (2010)
27. MadevaNagaral, V Auradi&Ravishankar M K, "Mechanical Behaviour of Aluminium 6061 Alloy Reinforced with Al₂O₃ &

- Graphite Particulate Hybrid Metal Matrix Composites*", *International Journal of Research in Engineering & Technology (IJRET)*, Vol. 1, Issue 2, pp. 193-198
28. Mohammad S. Alsoufi & Mohammed Yunus, "Effect of Heat Treatment on Stress Corrosion Cracking Resistance of Al-Zn-Mg-Cu Alloy Used in Aerospace Engineering Applications", *BEST: International Journal of Management, Information Technology and Engineering (BEST: IJMITE)*, Vol. 3, Issue 8, pp. 19-34
29. Nader H. El-Bagoury, Adel A. Omar & Ali A. El-Masry, "Effect of Semi-Solid Process on Microstructure and Mechanical Properties of Medium Carbon Steel Produced by Continuous Casting " *International Journal of Mechanical Engineering (IJME)*, Vol. 2, Issue 2, pp. 23-30
30. H. G. Mundle, "Variation in Strength of Concrete Subjected to High Temperature" *IMPACT: International Journal of Research in Engineering & Technology (IMPACT: IJRET)*, Vol. 2, Issue 2, pp. 149-154