Thermal Fatigue Cycle Shock Effects on Physical and Structural Properties of H13 Tool Steels before and after Heat Treatments

P. Karthikeyan¹ and Sumit Pramanik¹

¹Composite Laboratory, Department of Mechanical Engineering, SRM Institute of Science and Technology, Kattankulathur, Kancheepuram – 603203, Chennai, Tamil Nadu, India

E-mails: kp6423@srmist.edu.in, sumitprs@srmist.edu.in

Abstract. Any tool that is subjected to repetitive thermal service will undergo degradation in loss of various properties such as, strength, structures, thermal stability, and so on. These natural changes lead to huge premature failures and unexpected break downs during production by causing uneconomical and undesirable situations. Therefore in this present research, the raw, heat treated, and heat treated and nitrided H13 tool steel specimens were subjected to a thermal shock cycle condition similar to a real industrial application using a unique in-house built thermal shock cyclic fatigue (TSCF) testing machine to impose the thermal gradients. All the TSCF tested samples were then characterized by physical and structural tests, including, hardness, (Rockwell, HRc), X-ray diffraction (XRD), and field emission scanning electron microscope (FESEM). The interesting changes in hardness, distorted crystal structure, and crack initiation due to the imposed cyclic thermal gradients by TSCF process up to 2000 thermal shock cycles were found to be different for differently treated H13 tool steel specimens. Therefore, this present investigation specifically would help in predicting the design parameters and to fabricate the proper mould components of various casting products.

1. Introduction

Die casting technique has widely been used in industry owing to their high quality, low cost and short lead-time since last few decades [1]. Many components in different industries are employed under a high service temperature environment for a long period. For examples, the pistons of engines, the rotor in a steam turbine unit, and so on are continuously subjected to a force for long periods in a high service temperature environment undergo creep damage. As a consequence, their working life is deteriorated. Conventionally, the components which are repeatedly subjected to thermal stresses e.g., at the times of opening and closing of the moulds, starting and stopping of the turbine, starting and stopping of the automotive or aircraft engines, and so on result to the reduction in working life through various surface defects as well as metallurgical changes. Due to thermal gradients, all the high temperature components degrade their physical properties including, mechanical strength, structural, magnetic, electrical, and properties with service time. These deteriorations lead to early failure and unexpected break down during manufacturing in the industries [2, 3].

Thus, researches on die casting processes have extensively been reported to predict the duration life of die casting components. Thermal shock cyclic fatigue (TSCF) testing is a commonly used technique to investigate those simulated thermal shock studies [2-9]. There are several failure modes that limit the life and performance such as, erosion, corrosion, gross local adherence and fracture of the casting
alloy, particularly, in soldering. Formation of thermal fatigue cracks in aluminium and brass die casting is one of the crucial life-limiting tool failure mechanisms [10-12]. The thermal fatigue cracks are developed from the rapid alternations in die surface temperature, which may induce high stresses. The high induced stress can increase the plastic strain in the surfaces of tool or mould components during each casting cycle. The surface cracks may be generated within a few thousand cycles, or even earlier, depending upon the materials and processes due to the low-cycle fatigue range (<10^3–10^4 cycles) [11, 13].

Therefore, several materials such as, functionally graded materials (FGM) [6], cast iron [4, 7, 8], H21 steel [9], and H13 steel [5], have been carried out to undergo TSCF tests at low [4, 5] and high [6-8] thermal cycles for different applications. Particularly, the die materials are mainly made of hot worked tool steels, including AISI H11 [14], H13 [15, 16], H20 [11], H21 [17], H22 [18], and so on [11]. However in most of the cases, the premature failures are occurred due to thermal fatigue on die surface. It degrades the die quality and increases the cost of castings significantly. Therefore, many researchers have been trying to evaluate the effect of several surface treatments to prevent thermal fatigue crack formation and/or propagation of hot worked tool steels [11]. However, still there are many scopes to improve the thermal fatigue life of the components in many engineering fields owing to the different service conditions of such hot worked components and the complexity of thermal fatigue processes. In this context, H13 steel is very commonly used as tool steel and die casting mould components [1, 5]. Many researchers used laser surface modification, which can form a surface layer with strong metallurgical bonding to the tool steel’s substrate and acquire very rapid rates during surface melting. However, direct concentrated heating caused by the direct heating of specimens through laser power may damage the specimen’s surface, which is extremely significant rather than thermal shock. Therefore, it does not elucidate the purpose of thermal shock fatigue life prediction of the components in real applications. Therefore, this uncontrollable, direct heating surface damage difficulty can be overcome by this present invention. Here, the convection heating mechanism has advantageously ability to determine the thermal fatigue shock and subsequently evaluate the duration of thermal life of the specimens more precisely.

Therefore in this present study, the raw, heat treated, and heat treated and nitrided H13 tool steel specimens were subjected a thermal shock cycle condition similar to a real industrial application using a unique in-house built TSCF testing machine in order to impose the thermal gradients to the specimens.

2. Materials and Methods

The commercial grade H13 tool steels purchased from M/s Goel Steel were machined into proper specimens of size Φ10 mm × 100 mm for different heat treatment processes. The used heat treatments were two types: first is only hardening and second is hardening followed by nitriding in AD VAC Heat Treaters Pvt Ltd. In hardening processes, first raw H13 samples were preheated at 650 °C and 850 °C temperature for 30 min each and immediately heated to austenizing temperature at 1030 °C for 30 min in air atmosphere followed by oil quenching. In hardening and nitriding process, first raw H13 samples were preheated at 650 °C and 850 °C temperature for 30 min each and immediately heated at austenizing, 1030 °C for 30 min followed by nitrogen quenching and again tempered at 570 °C for three times in air atmosphere. Then, all the samples including, raw H13 specimens and heat treated specimens such as, hardening, and hardened and nitriding processed specimens were subjected to the thermal shock cyclic fatigue (TSCF) process. Here, both the heat treatments were done in industry but TSCF test was done using a newly developed unique TSCF testing machine. In the TSCF process, the specimens were kept in TSCF testing machine (see figure 1), which has provisions for variable cycle time, temperature and counting number of cycles. Entire methodology of the study is depicted in figure 2. Then, all the untreated and TSCF treated specimens of different size (Φ10 mm × 3-5 mm) were characterized by various tests, including hardness (Rockwell, HRc), density, X-ray diffraction (XRD), and field emission scanning electron microscope (FESEM).
3. Experimental

3.1. Density
Density of all the treat and untreated H13 steel specimens was measured by modified Archimedes’ principle using equation (1) using a weighing machine of resolution ±0.0005g as reported elsewhere [19, 20]. At least 3 identical specimens were used to calculate the mean density and their standard deviation (SD) of both the samples.

\[ \rho = \frac{W_1}{W_2} \times \rho_{\text{water}} \]  

where, \( \rho \) is density, \( W_1 \) is weight in air, \( W_2 \) is the weight in water, and \( \rho_{\text{water}} \) is density of water at room temperature (25 °C).

3.2. Hardness
Surface hardness of all the treat and untreated H13 steel specimens was determined by Rockwell hardness tester (model: RASNE-3, make: Fuel instruments and engineers (P) LTD, country: India)
using load 150 kgf load for 10 sec in HRC scale. At least 3 identical specimens were used to calculate the mean hardness of all the samples.

3.3. X-ray diffraction (XRD)

X-ray diffraction (XRD) study of all the treat and untreated H13 steel specimens was conducted in the range of diffraction angle, $2\theta = 15 – 90^\circ$ of Cu Kα radiation using x-ray diffractometer (model: X Pert Pro, make: PANalytical, country: UK) at normal scanning.

3.4. Morphology study by field emission scanning electron microscope (FESEM)

Morphology of all the treat and untreated H13 steel specimens was viewed under Field emission scanning electron microscope (FESEM) (model: QUANTA 200, make: FEI, country: USA) at different magnifications.

4. Results and Discussion

4.1. Density

Density of the untreated and TSCF treated specimens are illustrated in Table 1. The result shows that the average density of as received H13 steel increases from 7.527 g/cc to 7.731 g/cc for hardened H13 steel and further to 7.758 g/cc for hardened and nitrided H13 steel samples. It is because of more diffusion of carbon and nitrogen atoms into the interstitial positions of the steel after heat treatments. Density has also increased after TSCF treatment in hot chamber but decreases in molten metal immersion for all the three type samples (AR, HND and HN). The first result attributed to lowering of unit cell volume after treatment in hot chamber compared to the untreated AR, HND and HN samples. It was also confirmed by evaluation of lattice parameter from XRD study. The later result attributed to the sticky lowered density aluminum at the surface of the immersed samples.

4.2. Hardness

Average Rockwell surface hardness values of the samples are illustrated in the table 1. The average

| SAMPLES                          | Modified Archimedes’ Density (g/cc) | Hardness Before Thermal Shock Cyclic Fatigue Test (HRc) | Hardness After Thermal Shock Cyclic Fatigue Test (HRc) | Lattice parameter, $a$ (nm) |
|----------------------------------|-------------------------------------|------------------------------------------------------|------------------------------------------------------|----------------------------|
| RAW H13 Steel (AR)               | 7.527±0.619                         | 32±1                                                 | -                                                    | 0.2678                     |
| AR in hot chamber, HC, (AR-HC)   | 8.675±0.556                         | -                                                    | 32±1                                                 | 0.2647                     |
| AR immersed, IM, in molten aluminium metal (AR-IM) | 8.069±0.672                        | -                                                    | 34±1.2                                               | 0.2646                     |
| Hardened (HND)                   | 7.731±0.481                         | 48±1                                                 | -                                                    | 0.2672                     |
| HND-HC                           | 7.844±0.310                         | -                                                    | 46±0.9                                               | 0.2646                     |
| HND-IM                           | 7.415±0.385                         | -                                                    | 47±0.8                                               | 0.2641                     |
| Hardened and nitrided (HN)       | 7.758±0.714                         | 54±2                                                 | -                                                    | 0.2720                     |
| HN-HC                            | 8.699±0.740                         | -                                                    | 39±1                                                 | 0.2668                     |
| HN-IM                            | 7.893±0.603                         | -                                                    | 32±1.5                                               | 0.2664                     |
hardness (HRc) values of raw H13 steel were increasing from 32 to 48 for hardened H13 steel and it further increased to 54 for harden and nitrided specimen. It indicates that the average hardness (HRc) value reduces significantly in hardened and nitrided sample due to TSCF test. The deterioration of hardness in the TSCF treated hardened and nitrided samples (HN-HC and HN-IM) was high might be the cause of conversion of martensite to austenite by diffusion of interstitial nitrogen atoms from the solution. It was also confirmed by XRD study.

4.3. X-ray diffraction

XRD patterns of the raw, hardened, and hardened and nitrided processed specimens before and after subjected to the TSCF process are depicted in figures 3, 4, and 5, respectively. The basic phase of body centered tetragonal (BCT) crystal structure of martensite (α′-Fe) was observed for all the H13 steel samples. One of the main lattice parameters (a) for all the steel samples is illustrated in table 1. Values of lattice the parameters, ‘a’ closely resembled with the standard XRD data JCPDS No. 001-085-1410 and other study [21]. The lattice parameter decreased after TSCF treatment in hot chamber (HC) for the samples AR-HC, HND-HC and HN-HC compared to their untreated specimens. From the XRD results, it can be noted that the martensitic crystal structure parameters had been distorted due to TSCF test in all the TSCF tested samples (see figures 3b&c, 4b&c, and 5b&c). The (111) peak of Al appeared in all the TSCF tested immersed samples. It also indicates that more amount of sticky molten Al attached to the hardened and nitrited sample’s surfaces (see figure 5c). Interestingly, face centered cubic austenite (γ-Fe) phase also revealed after TSCF test of hardened and nitrited samples (see figures 5b&c). It is physically possible by proper heat treatments conditions as described by other researchers [22]. It happens due to the cyclic thermal treatments and as a result, some of the interstitial nitrides came out of the solid solutions and turned into γ-Fe phase.

![Figure 3. XRD of the raw H13 before and after TSCF test: (a) AR before TSCF test, and after TSCF test in (b) AR-HC and (c) AR-IM.](image-url)
Figure 4. XRD of the hardened H13 before and after TSCF test: (a) HND before TSCF test, and after TSCF test in (b) HND-HC and (c) HND-IM.

Figure 5. XRD of the hardened and nitrided H13 before and after TSCF test: (a) HN before TSCF test, and after TSCF tests in (b) HN-HC and (c) HN-IM.

4.4. Morphology study by FESEM

From FESEM result shown in figures 6a-i, it is clear that more distorted morphology can be obtained in the hardened, and hardened and nitrided samples after TSCF test. Surface crack and particle debonding were observed in both the heat treated samples figures 6(b,c,e,f,h,i). But, more amount of molten aluminium was attached to as received H13 sample’s surface, which is extremely undesirable for die components. It has also been supported by XRD study as shown by sharp large peak of (111) of Al in figure 3c for AR-IM sample. On the other hand, the hardened and nitrided samples showed more distorted surface morphology after TSCF test in hot chamber (see figure 6h) and also more Al at the surface of HN-IM sample (see figure 6i). Therefore at present TSCF conditions, the hardened and nitrided samples may enhance the crack initiation in hot chamber as well as in molten condition and hence, might decrease the life of die casting components.
5. Conclusions
Two different typical heat treatments were employed on the raw H13 specimens similar to the thermal cycles subjected to industrial components. The present results have shown that there are some changes in hardness, crystal structure distortion, and crack initiation due to the imposed cyclic thermal gradients by TSCF process at 670 °C up to 2000 thermal shock cycles. The results found to be different for differently treated H13 tool steel specimens. This present investigation predicts that the hardened H13 steel samples would delay the crack initiation hot chamber as well as in molten condition and hence increase the life of die casting components. Thus, the present thermal fatigue cycle up to 2000 numbers at 670 °C predicts that the present hardening heat treatment method would ultimately improve the life in aluminum casting service cycles and economical production of casting. Hence, present investigation may further be used to predict the design parameters and to fabricate the proper mould components of various casting products.

Acknowledgement
Department of Mechanical Engineering, NRC, Physics and Nanotechnology, SRM Institute of Science and Technology, Kattankulathur.

6. References
[1] Tong X, Dai M-j, and Zhang Z-h 2013 Appl. Surf. Sci. 271 373
[2] Vincent L, Poncet M, Roux S, Hild F, and Farcage D 2013 Procedia Eng. 66 669
[3] Sun J, Fu Q-G, Yuan R-M, Dong K-Y, and Guo J-J 2017 Materials & Design 114 537
[4] Tong X, Zhou H, Zhang Z-h, Sun N, Shan H-y, and Ren L-q 2007 Mater. Sci. Eng. A 467 97-103
[5] Zhang Z, Ren L, Zhou H, Han Z, Tong X, Zhao Y, and Chen L 2010 J. Bionic Eng. 7 390
[6] Fazarinc M, Muhič T, Šalej A, Bombač D, Fajfar P, Terčelj M, and Kugler G 2011 Procedia Eng. 10 692
[7] Tong X, Li F-h, Liu M, Dai M-j, and Zhou H 2010 Opt. Laser Technol. 42 1154
[8] Tong X, Zhou H, Chen W-w, Jiang W, Li X-z, Ren L-q, and Zhang Z-h 2009 *Opt. Laser Technol.* **41** 671
[9] Zhang Z, Lin P, Zhou H, and Ren L 2013 *Appl. Surf. Sci.* **276** 62
[10] Davis JR 1995 *ASM specialty handbook: tool materials* ASM international p 3
[11] Persson A, Hogmark S, and Bergström J 2005 *Surf. Coat. Tech.* **191** 216
[12] Persson A, Hogmark S, and Bergström J 2004 *J. Mater. Process. Technol.* 108
[13] Kovrigin V, Starokozhev B, and Yurasov S 1980 *Metal Sci. Heat Treat.* **22** 688
[14] Qamar SZ 2015 *Heat Treatment and Mechanical Testing of AISI H11 Steel* in *Key Engineering Materials* (Trans Tech Publ.)
[15] Karamiş M 1991 *Wear* **150** 331
[16] Telasang G, Dutta Majumdar J, Padmanabham G, and Manna I 2015 *Surf. Coat. Tech.* **261** 69
[17] Yan M and Fan Z 2000 *J. Mater. Sci.* **35** 1661
[18] Persson A, Bergström J, and Hogmark S 2002 Influence of surface engineering on the performance of tool steels for die casting *Proceedings of the 6th International Tooling Conference, Karlstad, Sweden* 1003
[19] Karthikeyan P and Pramanik S 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **402** 012037
[20] Pramanik S, Agarwala P, Vasudevan K, and Sarkar K 2019 *J. Appl. Polym. Sci.* **137** 48913
[21] Mazur J 1950 *Nature* **166** 828
[22] Bojack A, Zhao L, Morris PF, and Sietsma J 2016 *Metall. Mater. Trans. A* **47** 1996