DEVELOPMENT OF MARTEMPERED DUCTILE IRON BY STEP-QUENCHING METHOD IN WARM WATER

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Abstract: *The evaluation of the suitability of warm water for interrupted quenching of ductile cast irons in the production of Martempered Ductile Iron (MDI) was carried out. Ductile cast irons used were normalized at a temperature of 850^{o}C for 60 minutes. The normalized specimens were subsequently heat-treated in muffle furnace to a pre-determined temperature of 850^{o}C for 30 minutes, then step-quenched in warm water maintained at temperature of 80^{o}C for 40 seconds followed by tempering at different tempering temperatures (175^{o}C-425^{o}C) and times (30-180 minutes). Hardness test was used to evaluate the mechanical property while the metallographic examinations that produced different microstructures were used to evaluate the transformation behavior. The results showed that the developed MDI has a high average hardness value of 53Rc at the lowest transformation temperature, and 19.6Rc at the highest transformation temperature. Metallographic analysis showed that untempered martensite was obtained at holding temperature below 250^{o}C, tempered martensite at 250^{o}C to 325^{o}C, tempered martensite at holding temperature of 350^{o}C for short holding times, above which the specimen is over-tempered (depending on the holding time). This showed that warm water is a suitable quenchant for the production of MDI.*

Keywords: *martempered ductile iron, step-quenching, ductile iron, quenchant, interrupted quenching, process variables (Time and Temperatures).*

1. Introduction

Ductile iron, among other engineering materials, has found a great relevance in both structural and other related applications. Ductile iron (spheriodised or nodular cast irons) has the structure of graphite nodules in a matrix which may be ferritic, pearlitic or ferritic-pearlitic (ductile grades), bainitic (strong and tough grades) or martensitic (strong and hard grades). The variations in the matrix structure have caused the mechanical properties of ductile cast irons to vary widely and have made them applicable in a wide range of industrial applications such as automobile industry where high strength, toughness and ductility are required. The alloy elements such as silicon, chromium contained in ductile iron has been responsible for its difference with steel. The higher the silicon content, the lower the solubility of carbon in austenite and more readily carbon is precipitated as graphite during slow cooling to produce a ferrite matrix.

However, some researchers have been working on the improvement of the mechanical properties in ductile iron by heat treatment. This has led to the adoption of step-quenching method in a suitable medium to develop a MDI. Branka, et al. [1] and Hassan, et al. [2] studied the effects of some alloying elements in ductile iron and discovered that the addition of copper, nickel, or molybdenum to Austempered Ductile Iron (ADI) significantly improve the hardenability of the matrix and thus prevent the formation of pearlite during the austempering process. Larry[3] worked on several quenchants that could be used in step quenching processes, while the suitability of Khaha Senegalensis seed oil as a quenchant in austempering heat treatment of ductile iron was investigated by Isah et al.,[4]. All this has improved the utilization of ductile iron in automobile and machine tools industries as reported by Hemanth [5] work.

In attempt to reduce the high cost of production and extend the frontiers of knowledge in the production of MDI, this research tends to investigate the possible use of warm water for

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the interrupted or step quenching processes which is more user friendly and will minimize energy consumption that is associated with the use of salt-bath because of the elevated temperature of the salt as well as the increased capital equipment cost for the bath and the equipment that operates it.

2. Methodology

2.1 Materials and Equipment

The ductile iron specimens used for this research work were cast at Madison, United States of America (USA). The cylindrical specimens were cut into 20 mm diameter by 20 mm length dimensions. Other materials used are 2% natal solution (etchant) and desiccators. A mass spectrometric analyzer was used to analyze the chemical composition and was found to contain 3.6%C, 2.0%Si, 0.3%Mn, and 0.01% S as major elements. Metallographic equipment such as grinding and polishing machines, and optical microscope with camera of model AX10 (ZEIZZ) specification were also utilized for specimen metallographic preparations and examination. Other equipments used in the course of the experiment are muffle furnace, medium size lathe machine, digital Rockwell hardness tester Scale B (Averymodel) of (Rb 0 - Rb100), electric heater, mercury thermometer and stop watch.

2.2 Preliminary heat treatment

The specimens were normalized at 850^{o}C for 60 minutes. This was a preliminary heat treatment done to annul the mechanical history and to ensure compositional homogenization of the as-cast condition.

2.3 Production of Martempered Ductile Iron (MDI) by Interrupted Quenching

The normalized ductile cast iron samples were again austenitized at 850^{o}C, held for 30 minutes in muffle furnace and then quenched in warm water, which had been maintained at 80^{o}C, for a holding period of 40 seconds. After this, the samples were quickly transferred to another furnace (or electric oven of maximum temperature of 550^{o}C,), which had been maintained at a constant temperature of between 250^{o}C and 450^{o}C, for tempering process. The holding time for tempering in the furnace or oven was varied between 30 and 180 minutes after which the specimens were air cooled down to room temperature. Several specimens were treated per temperature for certainty. This test was done in accordance with Totten, et al [6] works.

2.4 Hardness Test

A digital Rockwell hardness tester Scale B (Averymodel) of (Rb 0 - Rb100) with a diamond indenter under the application of 150 KN load was used for the hardness evaluation in line with Jandin et al, [7] work. For each test specimen, a minimum of three points were measured and the averages were calculated.

2.5 Microscopic Examination

A Danheng software driven microscope of model AX10 (ZEIZZ) was used to analyze the microstructures of both the normalized and the developed MDI. Prior to this, the specimens were metallographically treated and etched with 2% natal solution, dried and then analyzed in line with the prior works of Davies and Oleman[8] and Guag[9].

3. Results

Table 1 contains the result of all the hardness measurements and Figures 1 and 2 for the plots of hardness against holding time at various tempering temperatures. The microscopic examination results showing the microstructures with the transformation behaviour of treated samples were as presented in Plates 1-8. The results were discussed under these headings.

3.1 (a) Hardness

Table 1 shows the hardness values for the ductile iron samples which have been subjected to interrupted quenching in warm water prior to holding at various times and various austenite transformation temperatures. It is seen that the hardness values varied between 52.9Rc, at the lowest transformation temperature, and 19.6Rc at the highest transformation temperature.

Table 1 Hardness Values Of Ductile Cast Irons Subjected To Interrupted Quenching In Warm Water Prior To Holding At Various Tempering Temperatures And Times.

TRANSFORMATION TEMPERATURES (^{O}C)	HARDNESS VALUES (R_c) FOR VARIOUS HOLDING TIMES (Minutes) TRANSFORMATION TEMPERATURES					
	30	60	90	120	150	180
175	52.9	51	48.8	47.2	45.5	42.9
200	46.0	42.9	39.8	40.7	37.3	33.6
225	43.8	40.1	39.0	36.7	34.5	32.9
250	42.2	38.7	35.9	33.6	30.7	28.9
275	35.9	35.4	33.0	32.1	29.8	27.3
300	38.7	36.5	31.7	30.8	28.6	26.9
325	32.7	31.0	30.1	29.9	28.0	27.2
350	33.6	29.7	28.6	27.4	26.7	26.2
375	31.8	31.0	29.7	28.2	27.6	27.7
400	37.8	27.2	26.1	26.0	25.9	25.2
425	26.6	23.6	23.1	21.2	20.5	19.6

From this result, it is expected that a wide range of martensitic structures at low transformation temperatures to high transformation temperatures will be found. This expectation is commonly found from specified hardness (R_c) values for different grades of ductile irons, based on the matrix structure, and specified in the ASTM A-536–70 standard.

Figures 1 and 2 show variation of hardness profile with both the holding temperature and time respectively, it is observed that the specimen treated at short holding times and especially at low austenite transformation temperatures possess a significantly high hardness values. This is confirmed by the predominantly martensite produced in the microstructures shown in Plates 1 and 2. This means that martensite structure was formed immediately after interrupted quenching in warm water and prior to transfer into the holding furnace. From the plots, an inverse relation was observed to exist between the hardness property and the tempering temperature of the developed MDI; this is because of relief of internal stresses and continued carbon diffusion, especially in the martensite structures that led to the reduction of the internal energy in the structures.

While some of the hardness profile shows essentially a continuously decreasing hardness at specific holding temperatures (when retained austenite is transformed to lower bainite), others, however, maintained unique curves characterized by:

i) An initial decrease in hardness with holding time indicating the retained austenite transformation.

ii) A bay of constant hardness, where there is short range carbon diffusion resulting in the formation of tempered martensite.

iii) A final decrease in hardness as a result of extensive carbon diffusion resulting in yet another tempered martensite in accordance with Janowak [10] and Harding [11] works.

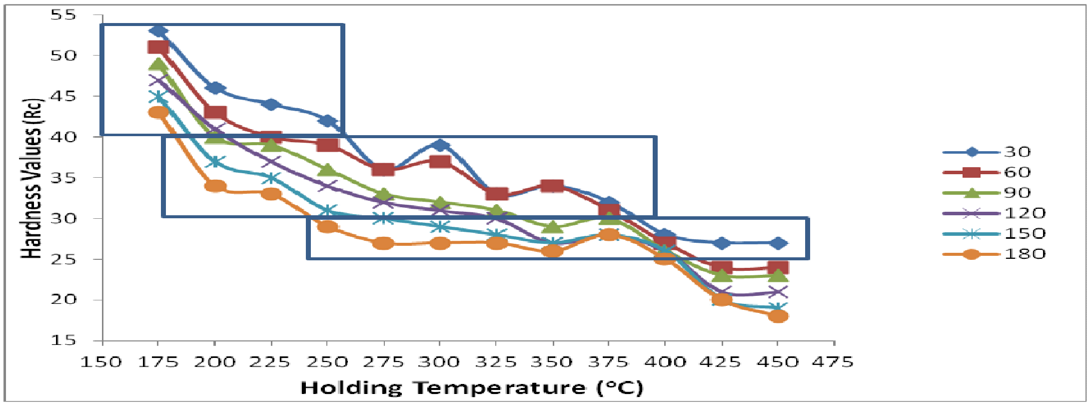

Figure 1: Variation of Hardness Values with Different Transformation Temperatures

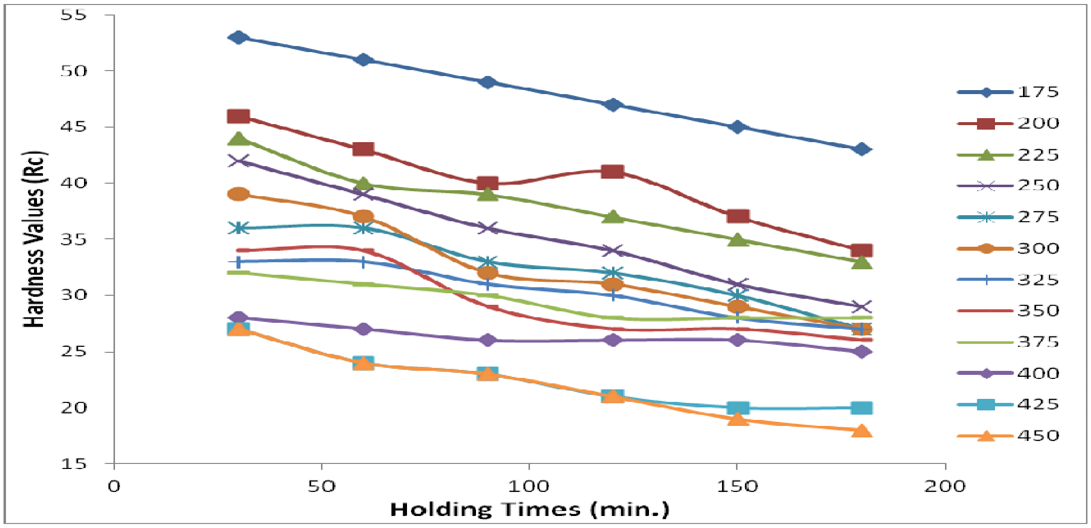

Figure 2: Variation of Hardness Values with Holding Times for Different Tempering Temperatures

3.2 (b) Microstructures

Plates 1 – 8 show the various developed tempered martensite structures. They are classified according to their holding temperatures. Plates 1 and 2 are treated at tempering temperatures between 175°C and 225°C. It is obvious that the structures obtained are essentially martensitic with some unavoidably retained austenite.

Plates 3–5 show the microstructures for ductile cast irons tempered at these tempering temperatures (250°C, 350°C, and 375°C) it shows structures which are typical of fine tempered martensite. Plates 6-8 consists of over-tempered martensite structure consisting of heavily spheroidized matrix structure with extensive ferrite shells surrounding the graphite nodules. Plate 6 shows spheroidized matrix structures with negligible ferrite shells, which occur for a short tempering period of 60 minutes at 425°C. However, prolonged heating for 120 minutes at low temperatures as shown in Plate 7 has been found to cause formation of thin ferrite shells around the graphite nodules. While Plate 8 shows that tempering at both high temperatures and for prolonged period of holding time, such as 120 minutes at 425°C, had resulted in the formation of spheroidized structure and extensive ferrite shells around the graphite nodules. The structure shows the existence of small seedlings consisting of ferrite and tiny spots of graphite at the centre. These seedlings are just the top parts of the full ferrite-pearlite nodules.

The structures obtained from the practical microstructural investigation correlate with the hardness predictions and are in agreement with findings in litratures of hardness study done by Ravishankar et al., [12] work. It has also confirmed that the above heat treatment process is realistic and reliable for the development of structures with improve mechanical properties comparable to those of austempered ductile irons.

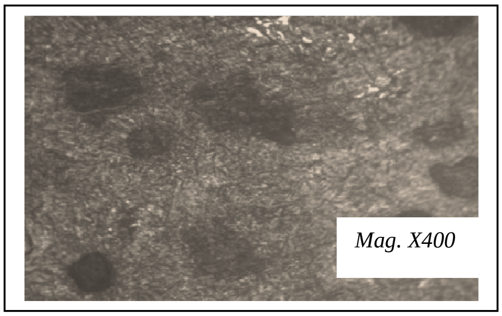

Plate 1: Ductile Iron tempered At 175°C For 30minutes showing martensite Structure.

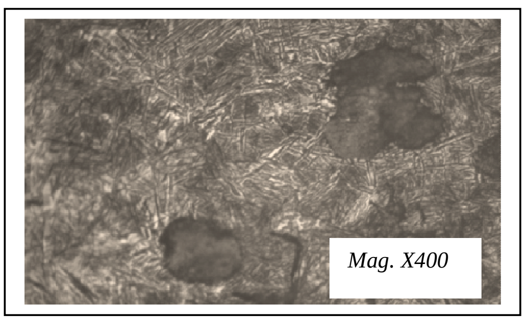

Plate 2: Ductile Iron tempered at 250°C for 90 minutes showing Type A tempered martensite structure.

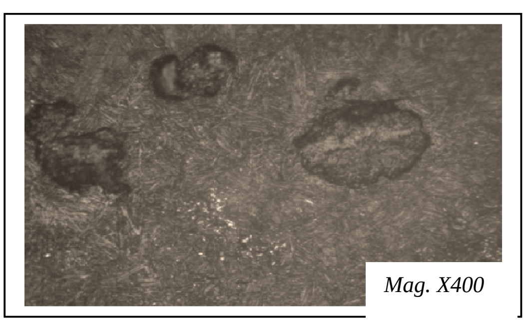

Plate 3: Ductile Iron tempered at 425°C for 60minutes showing spheroidized tempered martensite (dark) and cementite (white) structure.

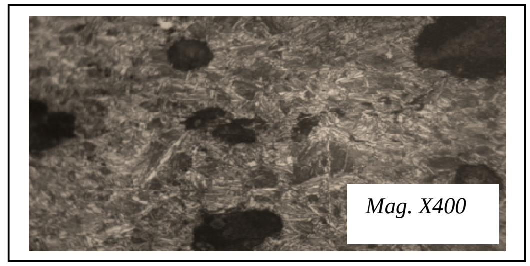

Plate 4: Ductile Iron tempered at 425°C for 60minutes showing spheroidized tempered martensite (dark) and cementite (white) structure.

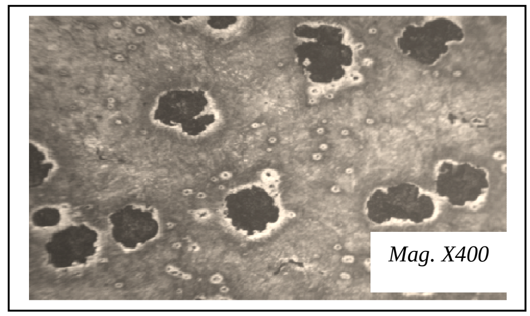

Plate 5: Ductile Iron tempered at 425°C for 60minutes showing spheroidized tempered martensite (dark) and cementite (white) structure.

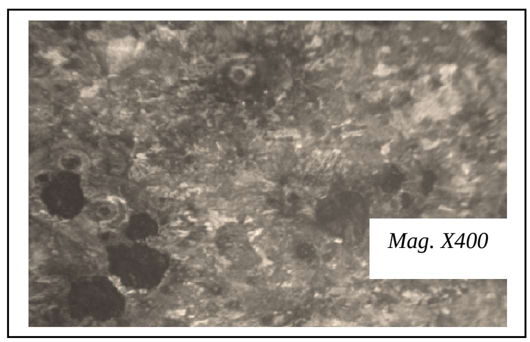

Plate 6: Ductile Iron tempered at 425°C for 60minutes showing spheroidized tempered martensite (dark) and cementite (white) structure.

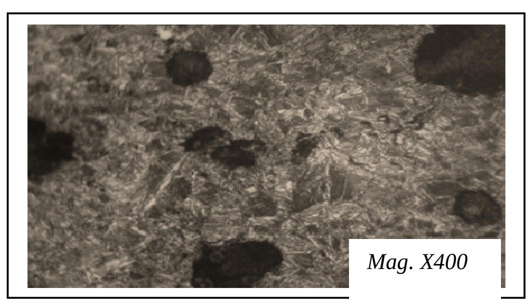

Plate 7: Ductile Iron tempered at 425°C for 60minutes showing spheroidized tempered martensite (dark) and cementite (white) structure.

4. Conclusion

Investigation of the suitability of warm water for interrupted quenching of ductile cast iron has been found to be successful. The ductile cast irons was austenitized at 850°C, subjection to interrupted cooling in warm water at 80°C for 40 seconds, tempered between the temperature range of 175°C and 425°C and a holding periods of 30 to 180 minutes before finally cooled in air. From the results, the followings were deduced:

(i) Warm water (cheap and readily available) could be used as quenchant in the production of MDI.

(ii) Martensite structure was formed after cooling in warm water for 40 seconds. Hence subsequent holding at higher temperatures resulted in tempering of the prior martensite structure.

(iii) Hardness values obtained after tempering ranged between 52.9Rc and 19.6Rc with an inverse relationship with holding temperatures and times.

(iv) A clear tempered martensitic structure was produced which had characteristics of martensite and bainites structures. At a temperature of ≥350°C over-tempering begins (depending on the holding time).

5. References

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