

## **Phase Transformation Study of Aluminium Containing Ductile irons by Dilatometry**

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The temperature for the start of martensite formation ( $M_s$ ) for three Al-alloyed ductile irons containing 0.48%Al, 4.88%Al and 6.16%Al were determined by high-speed dilatometry. Microscopic observations also were used to indicate the effects of aluminium addition on the martensite transformation during quenching. Various heat treatment cycles, with different continuous cooling and isothermal heat treatments, were used for the experimental irons to indicate the phase transformation in the solid state. The examination confirmed that an austenite/martensite transformation occurs for irons containing 0.48%Al and 4.88%Al. The results indicate that martensitic transformation occurs at about 200°C for 0.48%Al and about 190°C for 4.88%Al. Whereas there was no evidence of the martensitic transformation for 6.16% Al. It was further found that for the 4.88%Al experimental alloy used, isothermal transformation at 350° C for different soaking times gave a typical bainitic microstructure that increased with increasing time of austempering.

**Keywords:** Phase transformation, Dilatometry, Ductile iron, Aluminium, Martensite, Austenite

## **1. Introduction**

Higher tensile strength, better resistance to thermal shock, high graphitising tendency, good scaling resistance at high temperature and good machinability are some of the advantages which are obtained by adding Al to cast irons and many suggested uses include exhaust manifolds, turbo-charger housing, disk brake rotors, brake drum, cylinder liners, camshafts and piston rings. The satisfactory strength of Al-alloyed ductile irons provides the potential of this low-alloy ductile iron for elevated temperature service [1]. However, Al-alloyed irons have not been used fully and effectively in order to gain these advantages, because of difficulties encountered during casting. However, a clean casting can be obtained by correct design of the gating system which reduces turbulence in the mould and so prevents the inclusion of oxide films into the casting [1-5].

The exact appearance of the as-cast matrix microstructure of nodular cast irons, comprising ferrite, pearlite, cementite or a combination of these phases, depends upon chemical composition and cooling rate. A “bullseye” structure, which is spheroidal graphite surrounded by ferrite, is the most probable structure in unalloyed ductile irons. It is believed that the ferrite halo around the spheroid is formed before the eutectoid transformation. The volume fraction of ferrite increases as the cooling rate decreases, and fully ferritic structure is possible in heavy section casting [3]. Further heat treatment may cause more variety from formation of austenitic phase transformation products such as bainite, martensite or high-carbon retained austenite, which then confer different physical and mechanical properties on the iron [4].

It is apparent that the matrix in cast irons is virtually identical to that in steel. As for steel, the addition of alloying elements can modify the microstructure, and hence the physical and mechanical properties. As an example, the addition of copper refines the pearlite and promotes a

stronger and harder pearlite. The hardness of ferrite in the pearlite also can show an increase, due to solid solution hardening. Cu is a graphitising element and accompanied by Si suppresses the formation of hard carbides. The hardness continues to increase with the formation of pearlitic matrix as Cu increases and a substantial increase in tensile strength and wear resistance and excellent machinability also can be achieved. However, the low solubility of copper limits its application but it can be added with other elements such as nickel [6, 7].

Angus [1978] explained that for a 2 per cent addition of aluminium the pearlite formation temperature increases about 16° and 96° for 6.2%Al addition. Stefanescu and Martinez [8] showed that increasing the aluminium content in the Fe-C-Al system can decrease the level of intercellular carbides. It has also been illustrated that as the amount of aluminium in compacted/vermicular graphite cast irons in the Fe-C-Al system was increased, the pearlite content in the matrix also increased. It has also been reported that the microhardness of pearlite and ferrite increased with higher Al content, due to solid solution strengthening and a finer pearlite microstructure.

High-speed dilatometry results showed that the austenite reaction is related to the Al content and that there is no  $\gamma$ -loop at high Al concentrations; the austenitic reaction and therefore bainite reaction were not achieved for an iron containing 6.16%Al.

## **2. Experimental Procedures**

### **2.1. Melting**

The melts were cast with a pouring temperature of approximately 1350°C by both green sand moulding and gravity die casting. Standard 12.5 mm and 25 mm Y-block sand moulds and

permanent moulds were used and a bottom gating introduced the metal smoothly into the mould cavity to ensure a sound casting.

The experimental ductile irons with a range of composition were produced in a Morgan gas - fired furnace ( with 25 kg capacity lift - out crucible ) and a high - frequency melting plant of 20 kg capacity (with a tilting crucible). After melting, the iron was superheated at 1550°C and small pieces of solid aluminium added by plunging into the bottom of the liquid metal. Enough time was given to dissolve the aluminium completely in the molten metal. Following aluminium treatment, FeSiMg(5%Mg) alloy was plunged into the liquid iron and ejection of molten metal during solution of magnesium prevented by use of special enclosed reaction vessels. Finally, post-inoculation of ferro-silicon containing 75%Si was carried out in the crucible.

## **2.2. Chemical composition**

Quantitative measurements of the carbon content in the experimental irons were made using equipment at Swinden Technology Centre of Corus Group PLC (formerly British Steel Ltd.). Analyses were performed by an infrared absorption method after combustion in an induction furnace and oxidation to CO<sub>2</sub>. In order to analyse for aluminum in high-Al content ductile irons, an atomic absorption spectrophotometry (AAS) method was used by Hi-Search Technology (HIST) of Birmingham University.

## **2.3. Sample preparation**

Specimens 60x11x11mm for further experimental work were machined using an abrasive wheel cut-off machine type Cutmaster and a band saw type Qualters & Smith G10. The specimens were prepared from the bottom section of the experimental keel blocks (Y-blocks) in order to avoid

porosity and segregation effects in the ingots. A Struers Discotom-2 cutting machine was used for all optical and SEM samples. The specimens were then mounted in bakelite and ground to 1200 grit wet silicon carbide paper followed by polishing to 1 $\mu$ m with diamond paste. Phase identification was achieved after etching in 2% nital. The same techniques were used to prepare the specimens for hardness measurement.

#### **2.4. Hardness measurements**

After impact testing and examination samples with dimensions 20x10x10mm were cut from the Charpy specimens. Hardness measurements were made on these using a Vickers Engineering Group Vickers hardness machine at a load of 50 Kg on polished samples. A minimum of five measurements was made for each sample.

#### **2.5. Microstructural examination**

Optical microscopy and SEM were used to examine and delineate any variations in the microstructure e.g. the shape, size and distribution of nodular graphite, the size and distribution of bainitic packets and retained austenite, with changes in composition and heat treatment. Nikon and Reichert-Jung optical microscopes were used for optical metallography. The Nikon D.I.C (different interference contrast) microscope makes use of polarised light, incorporating polariser, analyser and compensating plate as in a normal polarised light microscope. To change the colours of the phases, the polarised dial is rotated until suitable contrast/colour was achieved. For scanning electron microscopy, a Cambridge Series 3 SEM fitted with a Link 860 series 1 EDX system and a Cambridge Series 4 SEM were used. For the characterisation of microstructure a working distance between 20-24 mm was chosen, and an accelerating voltage of 20kv and spot size between 4 - 6 nm.

## **2.6. Dilatometry**

Dilatometric experiments were carried out using a Theta Industries high-speed vacuum dilatometer (Dilatronic III) with rapid gas quenching. Cooling to the target temperatures was achieved by an automatically controlled high pressure helium jet. The tests were performed on 10 mm by 3 mm diameter tubular specimens with a 1.5 mm diameter bore in the centre to enable fast quenching. 0.15 mm thermocouple wires were spot welded to the specimens to measure variation of temperature during the heat treatment cycles.

Various heat treatment cycles were carried out for different conditions of continuous cooling and isothermal heat treatment to define the solid-state phase transformations in the experimental irons. After austenitising at different temperatures specimens were gas quenched with helium at different cooling rates to achieve the best condition for continuous or isothermal heat treatments.

Determination of the solid-state transformation in an iron during heat treatment with different cooling rates and various isothermal temperatures and different holding times is associated with deviations from linearity in length - temperature curves. To calculate the solid-state phase transformation temperature, it is assumed that volume changes of the samples are affected by the amount of transformation. In addition, metallography is used to determine or verify the relationship between heat treatment cycle and phase transformation.

### **3. Results and Discussions**

The influence of heat treatment conditions and cooling rate on transformation in three kinds Al-alloyed iron in a range of 0.48% to 6.16% Al has been investigated. Evidence shows that martensitic and bainitic transformations are possible in cast iron containing Al in the approximate range 0.48% to 4.88%. The transformation temperatures and morphology of these products are discussed.

The temperatures for the start of martensite formation ( $M_S$ ) for three Al-alloyed ductile irons were determined by high-speed dilatometry. Optical microscopy and hardness measurements also were used to indicate the effects of aluminium addition on the martensite transformation as reflected by the  $M_S$  obtained during quenching. For this study, specimens were heated at different austenitising temperatures where austenite grain growth can occur. After sufficient holding time, the samples were quenched to room temperature at different cooling rates. During cooling austenite is transformed to martensite and due to the transformation volume change the  $M_S$  is identified by deviation of the expansion/contraction curve from linearity. Figure 1 indicates the martensite reactions detected by high-speed dilatometry for a cooling rate of  $13^\circ\text{C/s}$ ; the curves

also show the variation of  $M_s$  with aluminium content. The results indicate that martensitic transformation occurs at about 200°C for 0.48%Al and 190°C for 4.88%Al (Figures 1b and 1d). Dilatometric examination carried out by Bhadeshia and Edmonds [9] showed that the martensitic transformation for a silicon steel started at 220°C. Based on phase transformation studies, it is suggested that aluminium additions decrease the activity of carbon in austenite and its effect on further transformation and the  $M_s$ . Bhadeshia [10] reported that depression of the martensite start temperature is possible by solute additions which decrease the driving force for the decomposition of austenite and retard the phase transformation rate.

On the other hand, for 6.16%Al a large difference is found amongst the experimental irons. The examination confirmed that an austenite/martensite transformation occurs for irons containing 4.88%Al and less (Fig. 2), whereas there was no evidence of the martensitic transformation for 6.16% Al (Fig 1f). The results thus indicate that 6.16%Al ductile iron is different from the lower Al content irons tested. The difference in transformation probably arises because there is no  $\gamma$ -loop for the higher Al content in the Fe-C-Al-Si system [4].

It was further found that for the 4.88%Al experimental alloy used, isothermal transformation at 350° C for different soaking times gave a typical bainitic microstructure that increased with increasing time of austempering [4]. Before isothermal heat treatment, the specimens were austenitised at 920°C. Following austenitisation, specimens were quenched to 350°C with a high cooling rate (50°C/s) to prevent any transformation before austempering at this temperature for different holding times.



The microscopic observations made in the present work illustrated that even for the short isothermal transformation times used (30 sec at 350°C), bainitic ferrite has already been formed. However, it should be noted that for times less than 1min, there is not enough deviation from linearity on the dilatometric curves that could be used to indicate the bainitic transformation. The remaining austenite will be changed to martensite during cooling to room temperature as demonstrated clearly in the related length-temperature curves [4]. In this case metallography is also needed to follow the phase transformation for each iron tested [11, 12].

#### **4. Conclusions**

The following results were determined by high-speed dilatometry, phase diagram study, optical microscopy and hardness measurements for different Al-alloyed ductile irons:

- Martensitic transformation occurs at about 200°C for 0.48%Al and 190°C for 4.88%Al, whereas there was no evidence of martensitic transformation for 6.16%Al, due to the fact that there is no  $\gamma$ -loop at 6.16%Al in the Fe-C-Al-Si system.
- Isothermal transformation for the 0.48%Al and 4.88%Al at 350°C for different soaking times confirmed that a typical bainitic microstructure is formed that increased with increasing austempering time.

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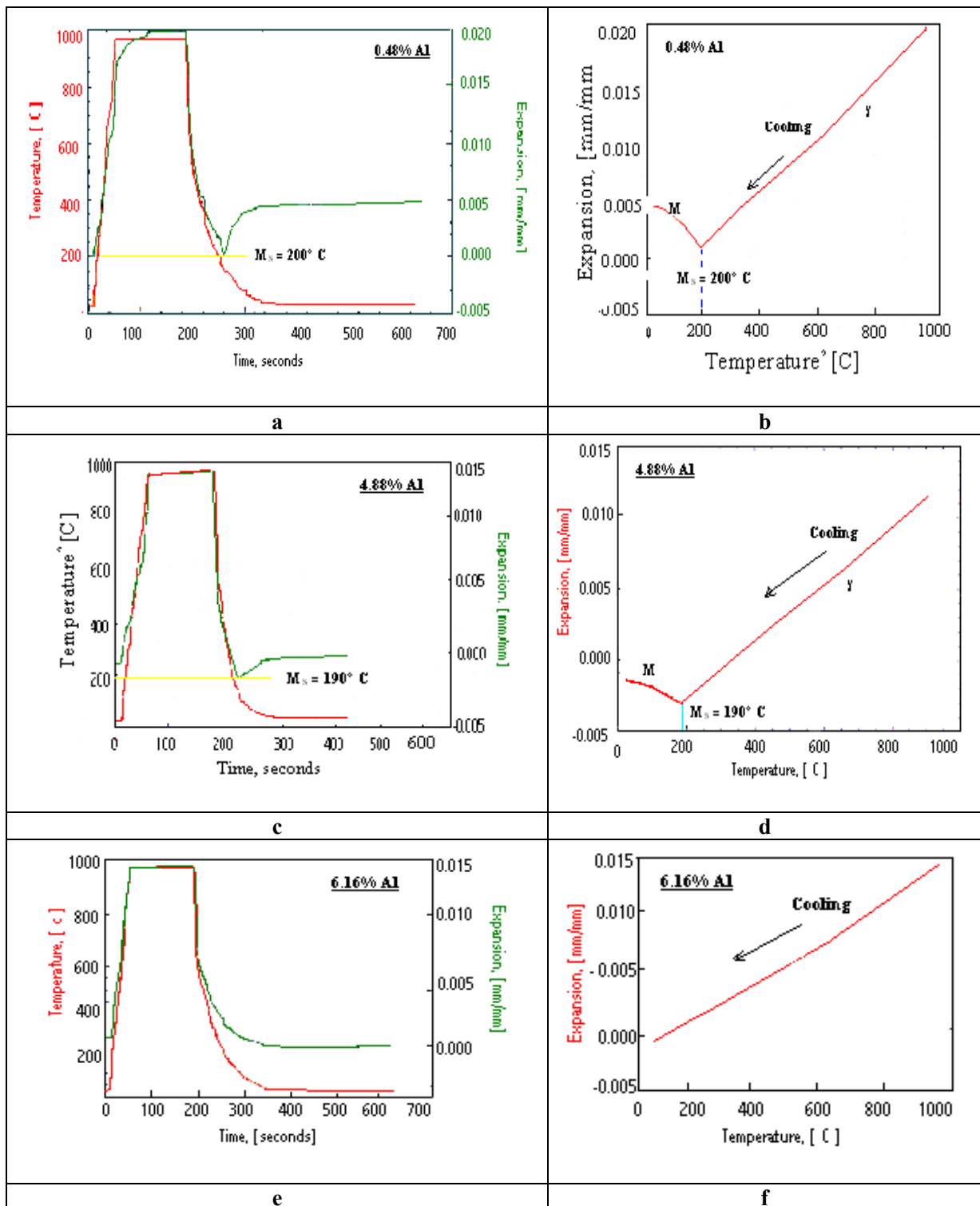
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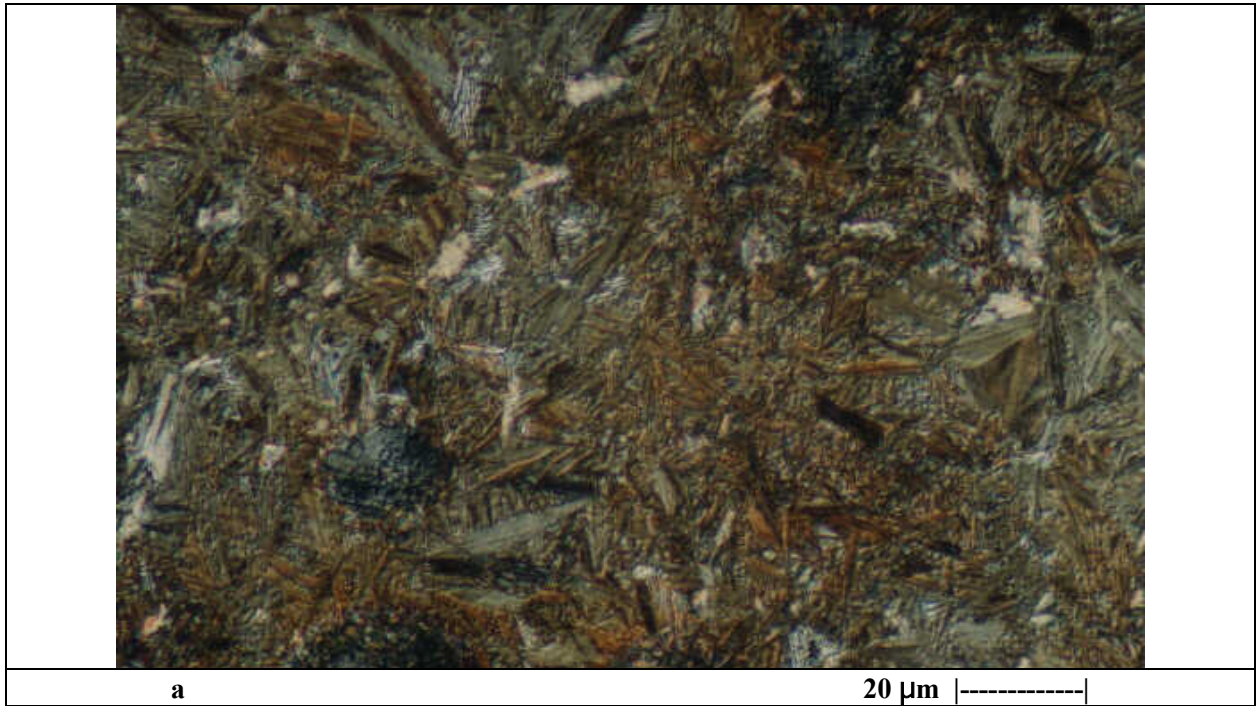
## 7. Tables and Figures

**Table 1** Composition of irons, wt-%

<b>Alloy</b>	<b>C</b>	<b>Al</b>	<b>Si</b>	<b>Ni</b>	<b>Mn</b>	<b>P</b>	<b>S</b>	<b>Mg</b>	<b>Fe</b>
0.48%Al	3.68	0.48	1.06	0.04	0.06	<0.005	<0.005	0.05	balance
4.88%Al	3.44	4.88	1.22	0.05	0.10	<0.005	<0.005	0.05	“
6.16%Al	3.25	6.16	1.35	0.07	0.10	<0.005	<0.005	0.06	“



**Figure 1** The heat treatment cycles used for ductile irons; a) 0.48%Al, c) 4.88%Al and e) 6.16%Al and the corresponding expansion versus temperature plots for ductile irons; b) 0.48%Al, d) 4.88%Al and f) 6.16%Al



**Figure 2** The typical martensitic transformation of Al-alloyed ductile irons austenitised at 920°C and cooling to the target temperatures by using an automatically controlled high pressure helium jet.