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Investigations on the suitability of some ferrous alloys for semi-solid processing

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ABSTRACT

Purpose: the work analyses the thixoability of SAE 1070, SAE 1548 and SAE 4340 steels; the possibility of producing thixotropic semi-solid by partial melting of these alloys and the phase transformations taking place during the process.

Design/methodology/approach: thixoability was characterized by differential scanning calorimetry (DSC) to determine transformation temperatures involving liquid formation and df_l/dT within the solidification range. Thixotropic slurries were produced by heat treatments at different temperatures above T_s, and distinct holding times. Microstructures were analysed by RX diffractometry, optical and electronic microscopy and EDS microanalysis.

Findings: results show that the three alloys investigated present high thixoability, given by wide solidification ranges associated with affordable sensitivity of liquid fraction with temperature within these ranges. Higher thixoability is presented by SAE 1070, followed by SAE 1548 and SAE 4340. Results show also that is perfectly feasible the production of thixotropic slurries of all investigated alloys, by simply heating to temperatures where a liquid phase can be present. Spheroidisation of solid primary phase is fast and increasing holding time at the semi-solid temperature leads to excessive growth of the globules in the thixocast material.

Research limitations/implications: thixoability prediction models rely on sensitive experiments as thermoanalysis, with results strongly dependent on experimental conditions; and on thermodynamic data, sometimes not available or reliable for a specific alloy composition.

Practical implications: the prediction of the thixoability of a certain alloy can make it more effective its thixoprocessing, allows better control of processing parameters and of the quality of final product. The viability of producing thixotropic semi-solid of ferrous alloys by simple partial melting can definitively insert these families of metallic alloys in the semi-solid processing field.

Originality/value: ferrous alloys have become part of the thixoforming scenario more recently, when compared to aluminium and magnesium alloys. Therefore, the study of the thixoability of three commercial steels can bring important information as far as their utilization in thixoforming processing is concerned.

Keywords: Casting; Thixoability; Thixoforming; Semi-solid alloys

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1. Introduction

Processing of thixotropic semi-solid metallic alloys has been increasingly present in the market for the production of near net shape components, mainly in the automotive industry. In the last 3 decades, semi-solid processing area has been occupied almost exclusively by aluminium alloys and, in less extend, magnesium alloys. However, the development of techniques to produce thixotropic semi-solid from solid material avoiding the need to handle liquid metal, allowed the insertion of high melting point alloys in the SSM scenario. In fact, thixoforming of different types of high alloyed steels has been lately investigated [1,3], as well as also some cast iron [4]. Moreover, with the progress of the semi-solid technology development along the years and the consequent familiarization with the control parameters of the thixotropic slurries fabrication and forming processes, it was possible to establish some basic requirements for a specific alloy to be candidate to thixoforming processing. The thixoability of an alloy must gather some criteria such as the solidification range, the liquid fraction at eutectic temperature, and the sensitivity of liquid fraction with temperature [5,6,16]. The first criteria can be limited by the susceptibility of the alloy to hot cracking during cooling if the solidification range is too wide; concerning the second criteria, it has been observed that more controllable is the processing if eutectic reaction occurs at 30 to 50% liquid and third criteria states that df_l/dT can not be too abrupt, otherwise little changes in temperature can lead to big variation in the liquid fraction and make it difficult to control the process. Results for Al-Si-Cu alloys [7,8] indicate values of df_l/dT between 0.007 K^{-1} to 0.067 K^{-1} at f_l=0.4, depending on the Si-Cu content. It means that an increase of 1K in the alloy presenting 40% liquid implies an addition of 0.7 and 6.7 in the % of liquid content in the semi-solid; thixoability is higher only in the first case, allowing easier manipulation of the alloy in the semi-solid state. Data on thixoability of ferrous alloys indicate low thixoability for ASTM A536 cast iron, due to a narrow thixoforming window (around 30°C) and a high sensitivity of liquid fraction with temperature within this range (0.056°C^{-1} at f_l = 0.45) [4]. Also some special classes of steels, such as stainless and tool series have been catching attention and some results on their thixoability are available [9-11].

The work analyses the thixoability of three steels grades, SAE 4340, SAE 1070, and SAE 1548 which find commercial application in the automotive industry as several cast components; and the actual viability of production of thixotropic semi-solid by partial melting. The viability of application of semi-solid technology for ferrous alloys can bring a whole new concept on forming complex shapes at low energy consumption, not available today due to the high melting temperatures of these alloys and the high energy required for their mechanical forming.

2. Experimental procedures

Commercial SAE 1070, SAE 1548 and SAE 4340 steels were used in the work; their chemical compositions are presented in Table 1. Those steels find general application for components that require good mechanical properties in large sections and are widely employed in a series of mechanical components as crankshafts, gears, shafts, etc.

Table 1.
Chemical composition of SAE 1070, SAE 1548 and SAE 4340 steels (wt%) used in this work

SAE Designation	C	Mn	P	S	Cr	Si	Ni	Mo
1070	0.70	0.65	0.01	0.067	0.14	0.20	0.06	0.03
1548	0.46	1.21	0.021	0.023	0.14	0.22	0.01	0.04
4340	0.41	0.69	0.01	0.009	0.82	0.27	1.77	0.24

The three alloys are hypoeutectoid systems; SAE 1070 and SAE 1548 steels present at room temperature cast structures containing pearlite and α ferrite, while SAE 4340 can present martensite and ferrite, as high Cr and Ni contents can prevent eutectoid transformation, as austenite stabilizers, increasing the material temperability.

The experiments were divided in two series: study of the thixoability of the considered alloys and effective production of samples of thixotropic semi-solid for observations of microstructures in the thixocast condition.

For thixoability studies, differential scanning calorimetry (DSC) tests were performed, using heating cycles at the rate of 10°C/min. From resulting curves, temperatures of phase transformations, mainly those involving liquid formation were obtained; f_l x T curves were built to evaluate the sensitivity of liquid fraction with temperature in the solidification range, for the alloys considered.

Samples of thixotropic semi-solid were produced by partial melting of as-cast material (SAE 1070 and SAE 1548) and of forged material (SAE 4340), at different temperatures and holding at a constant pre-defined temperature for 0 and 20min followed by rapid cooling in water. Temperatures used varied from 1455 to 1475°C for SAE 1070 steel and from 1460 to 1485°C for SAE 1548 and SAE 4340 steels.

Conventional metallography technique was used to prepare cross sections for microstructure observations; Nital 2% was the etching employed. X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM) as well as microanalysis by EDS in specific regions, were used to characterize microstructures.

3. Results and discussion

3.1. Thixoability analysis

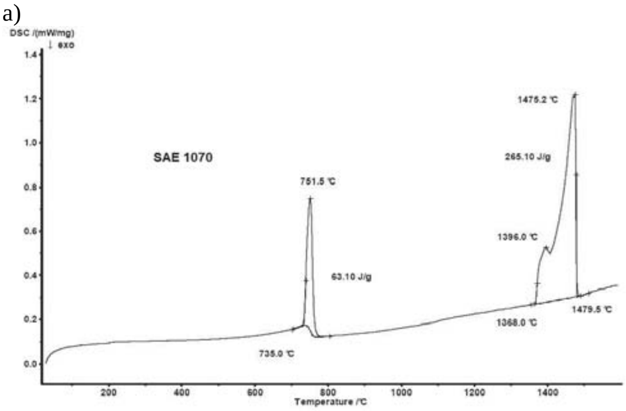
Resulting curves of thermal analysis by DSC of the studied alloys are presented in Figures 1 (a-c), respectively for SAE 1070, SAE 1548 and SAE 4340 steels.

It can be clearly observed the presence of two main peaks, in all cases, which can be related to the solid-liquid transformation, and the eutectoid transformation (the latter around 750°C in all cases).

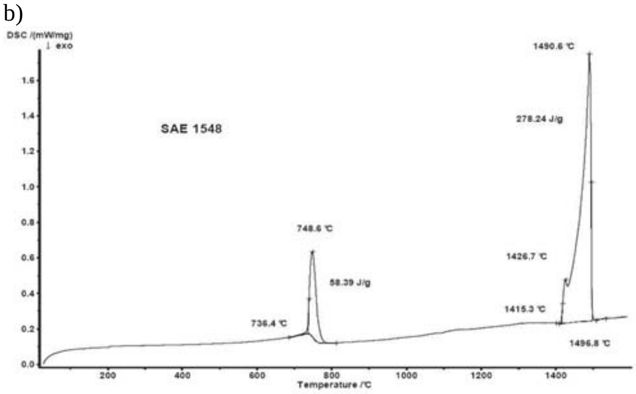
Peaks at higher temperature shall include L + δ and L + γ fields, as observed in the general phase diagram for Fe-C binary system presented in Figure 2. On cooling from liquid, in the case of SAE

1070 steel it is expected the formation of γ phase directly from the liquid as primary phase, while for the steels with lower C contents, the primary phase forming at higher temperatures, would be δ ferrite. Therefore, in the semi-solid material the solid component is always γ for SAE 1070 and can be either γ or δ for SAE 1548 and SAE 4340, depending on the temperature. On cooling from semi-solid region, in the case of steels with δ ferrite as primary phase, C content in the liquid is increased until a peritectic reaction takes place originating the γ phase. If equilibrium conditions are reached, all δ phase is consumed in the transformation.

a)



b)



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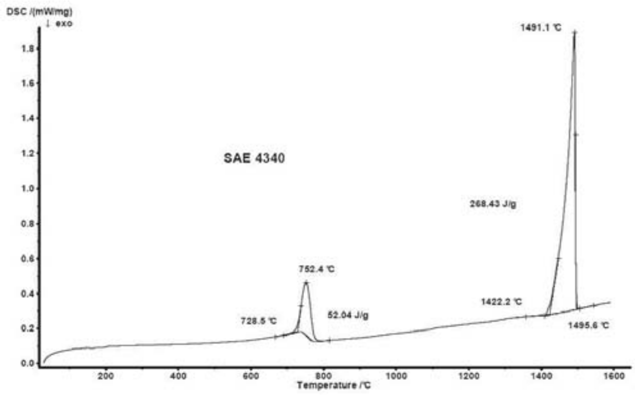


Fig. 1. DSC curves of the studied steels: a) SAE 1070, b) SAE 1548 and c) SAE 4340. Heating rate: 10°C/min

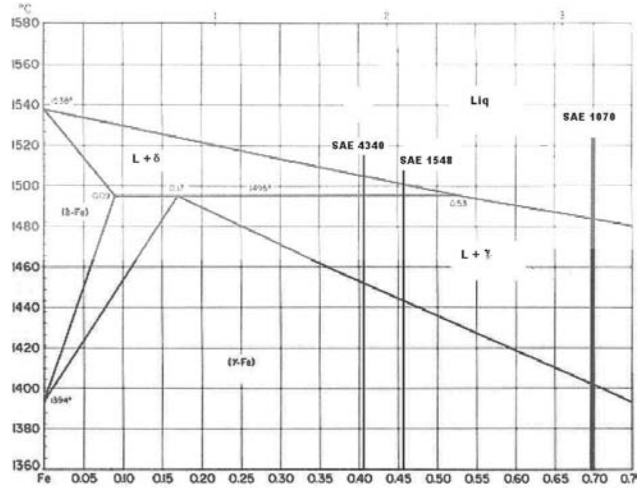


Fig. 2. General phase diagram for Fe-C binary system [12]. Fe-C compositions of investigated steels are indicated

According to the general Fe-C phase diagram and temperatures indicated in the DSC curves as beginning and finishing of the main peaks assumed to be related to the solidification transformation, the three steels considered show a considerable solidification range.

As far as thixoforming is concerned, not only an appropriate temperature interval must be available to allow handling of the semi-solid material during processing but, most important is the sensitivity of liquid fraction with temperature within this specific range. Moreover, the lower the temperature where appropriate viscosity for the semi-solid is achieved, lower the energy costs on heating up the material. Therefore, in the cases of the studied alloys, temperatures do not need to be as high as the peritectic temperature. Most of the S + L range lies in the region where the γ phase is the solid present. Therefore, it is expected that the thixotropic semi-solid will contain γ globules in the liquid, for all investigated steels.

However, the thixoability of the alloys were evaluated taking in account the whole S + L range, it meaning from T_s ($f_l = 0$) when, on heating up the material, liquid starts to form by melting of γ phase, to T_l ($f_l = 1$) when all γ has melted (for SAE 1070 alloy) and all δ phase has melted (cases of SAE 1548 and SAE 4340 steels). Therefore, the variation of liquid fraction with temperature was calculated from obtained DSC curves taking the area defined by the whole solidification peak. For a specific temperature within the beginning and the finishing of the transformation L → S, the liquid fraction is given by the relative area:

$$f_1(n) = \frac{\text{Partial area An (Tn - Ti)}}{\text{Total area At (Tf - Ti)}} \qquad (1)$$

where f_l (n) = liquid fraction at a generical T_n , is the ratio between the area under the DSC curve limited by T_n and the temperature where liquid fraction is 0 - the temperature where transformation initiates (T_i) - and the total area under the peak limited by temperatures where transformation initiates and finishes (T_f).

Results are shown in Figure 3. Acceptable processing window for thixoforming is generally considered between 20 to 60% liquid. Processing window is the range between maximum and minimum liquid fraction where semi-solid can be reliably processed. In casting operations liquid fractions around 0.6 can be quite feasible, while for mechanical forming operations sometimes liquid fractions as low as 0.05 must be the limit, as for instance in drawing of Al alloys sheets in semi-solid state [13].

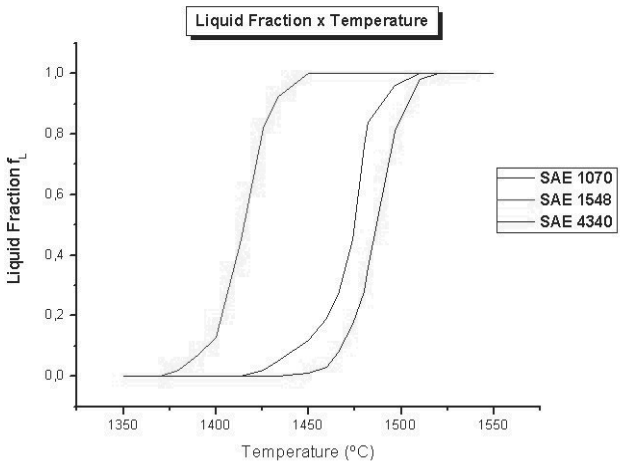


Fig. 3. Variation of liquid fraction with temperature within solidification range of SAE 1070, 1548 and 4340 steels

Figure 3 shows that the variation of liquid fraction with temperature is not linear: it is lower at the beginning and finishing of the transformation; the slope of the curve is higher exactly within the processing window. Therefore, it is clear that the material thixoability depends on the sensitivity of df_l/dT at processing temperatures: liquid fraction shall not change drastically with temperature, otherwise process control can become critical and even impossible.

Table 2 shows transformations temperatures given by the tangent to the DSC curves at the inflexions attributed to the solidification transformation, as well as the sensitivity of liquid fraction with temperature at a specific liquid fraction ($f_l = 0.5$).

Table 2.
Characteristic transformation temperatures and slopes of f_l x T curves for the investigated SAE 1070, SAE 1548 and SAE 4340 steels

SAE designation	T_s (°C)	$T_{0.5}$ (°C)	T_1 (°C)	$T_1 - T_s$ (°C)	df_l / dT at $T_{0.5}$ (°C^{-1})
1070	1368.0	1454.7	1479.5	110.5	0.00576
1548	1415.3	1479.5	1496.8	81.5	0.00779
4340	1422.2	1480.6	1495.6	73.4	0.00856

It can be observed that both the range of coexistence of liquid and solid, and the variation of liquid fraction with temperature are quite distinct for the three steels considered. SAE 1070 presents the wider solidification range and the smallest value of df_l/dT at the temperature equivalent to 50% liquid. In this situation, if a semi-solid with $f_l = 0.5$ has its temperature increased by 1ºC, the liquid fraction will increase 0.00576. For the other steels investigated, also wide solidification ranges are present and values of df_l / dT are perfectly affordable for SSM processing.

For comparison, previous studies on ASTM A536 cast iron show values of $df_l/dT = 0.056C^{-1}$ for $f_l = 0.45$ [4] as mentioned previously; such high sensitivity of liquid with temperature indicates the need of a close control of thixoforming temperature to insure the viability of the process for this kind of alloy. On the other hand, for Al-Si and other Al alloys already widely commercially employed for semi-solid processing, df_l/dT ranges from 0.007ºC^{-1} to 0.07ºC^{-1} at f_l=0.4. As far as steels are concerned, df_l/dT values from 0.0097 to 0.02 are reported for Cr-Mn alloy steels [10].

Therefore, results obtained show that the three investigated steels present high thixoability, given by a solidification range lying from around 70 to 110ºC and low sensitivity of liquid formation with increasing temperature within solidification range. Comparing the three steels studied, SAE 1070 steel presented the best conditions for semi-solid processing, followed by SAE 1548 and SAE 4340, in this decreasing thixoability sequence.

3.2. Thixocast microstructures produced by partial melting

As the three investigated alloys showed potential ability for thixoforming, semi-solid samples were produced for structure observations, by partial melting of as-cast or forged materials, at temperatures within the pre-determined solidification range, at different temperatures. Effect of holding time was also observed. Results are shown as follows.

SAE 1070

Typical resulting microstructures of SAE 1070 steel produced by heating the material at $T > T_s$ are shown in Figure 4, for samples heated to 1470ºC and chilled as it reached the treatment temperature and after holding for 20min at this temperature. It is also presented the initial condition of the material (as-cast) and the RX diffraction pattern of each condition.

As the steel is a hypoeutectoid system, with C content very close to the eutectoid composition, its structure in the as-cast condition is formed, at room temperature, essentially by eutectoid pearlite, with some pro-eutectoid ferrite in pearlite boundaries. RX diffraction shows massive presence of ferrite. The total amount of cementite in the system would be, in equilibrium conditions, around 10%; such small amount could explain the absence of cementite in the diffraction peaks.

Samples treated at lower temperatures (not presented here) show also globular structures, however with worse definition of boundaries due to the lower liquid content in the semi-solid.

After heating at $T>T_s$ the material presents globular structure as expected in typical thixocast materials. The globules are constituted by martensite, as indicated by XR patterns, as a result of the rapid cooling imposed to the thixotropic semi-solid from treatment temperature, where the primary phase present in the liquid was austenite. Some remaining austenite was also detected in the final structure, however in small amount. Interglobular region, which was liquid in the semi-solid condition, present MnFe and MnMoFe rich precipitates according to EDS analysis; probably some FeS as well. It can be observed that globularization of austenite grains in the liquid at high temperature is fast, once the morphology of original pearlite grains is already near globular: no holding time at the treatment temperature is required to produce globular particles in the semi-solid.

Globules in the thixocast material present dimensions of same order of magnitude of the initial pearlite grains they originate from, in the case of nil holding at the semi-solid temperature; globules diameter varies from 30 to 80μm, which can be considered appropriate for thixocast material and can lead to good rheological properties and mechanical properties in the final product. The globularization phenomena involved are coarsening mechanisms such as ripening, thermodynamically driven by the need to reduce superficial energy. At high temperatures, mass transportation is fast, so are diffusion mechanisms, leading to globular morphology of the primary phase in short time. On the other hand, this high kinetics can result in excessive growth of the solid phase as holding time is increased, as observed in the microstructure held for 20min at the treatment temperature. As far as thixoforming is concerned, globules of big dimensions are not desirable if good rheological properties are required.

a)
b)
c)

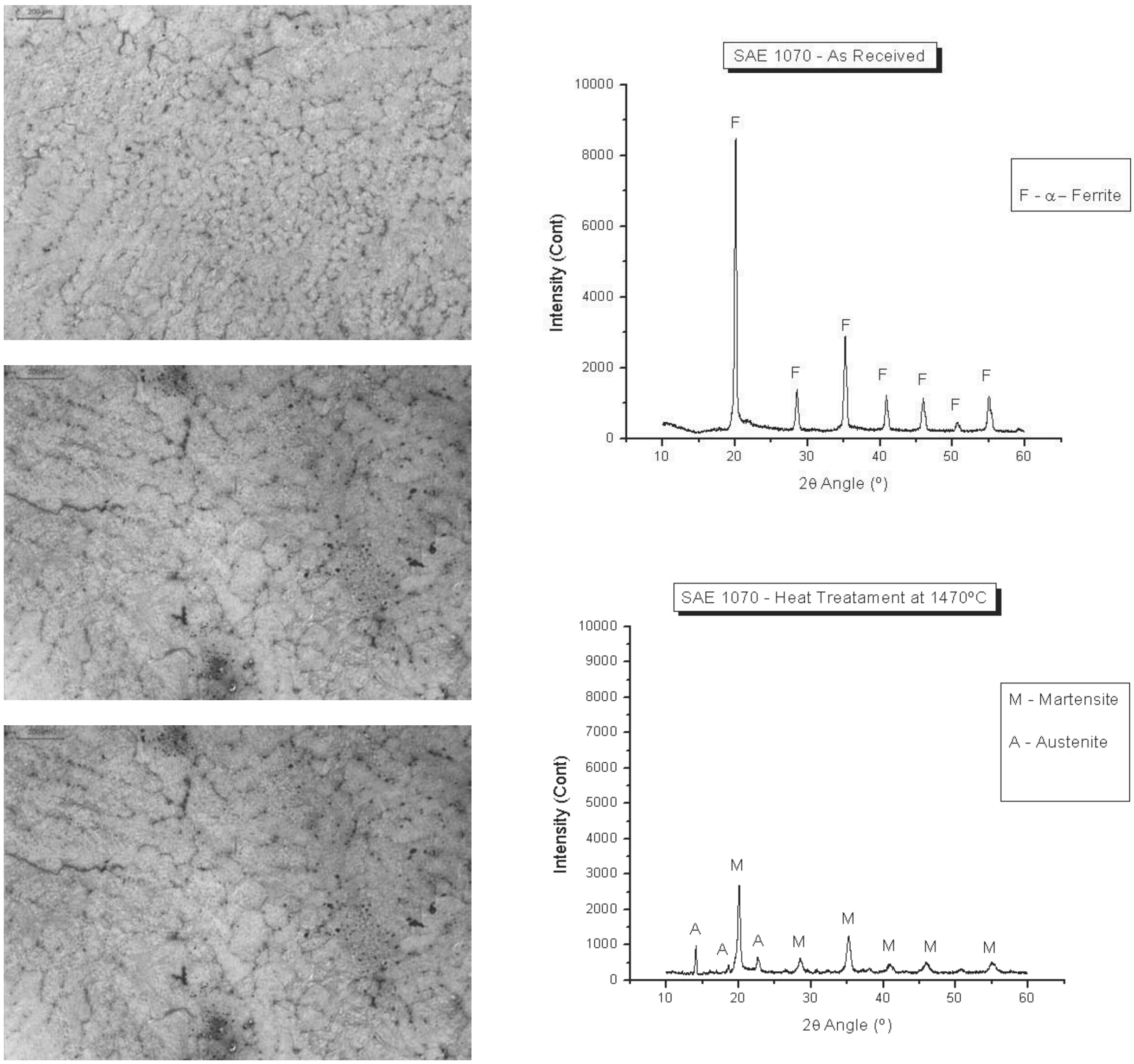


Fig. 4. Microstructures of SAE 1070 steel (optical microscopy) and correspondent XRD patterns, in different conditions: a) as-cast, b) after heat treatment at 1470ºC, 0 min, c) after heat treatment at 1470ºC, 20 min. Quenched in water

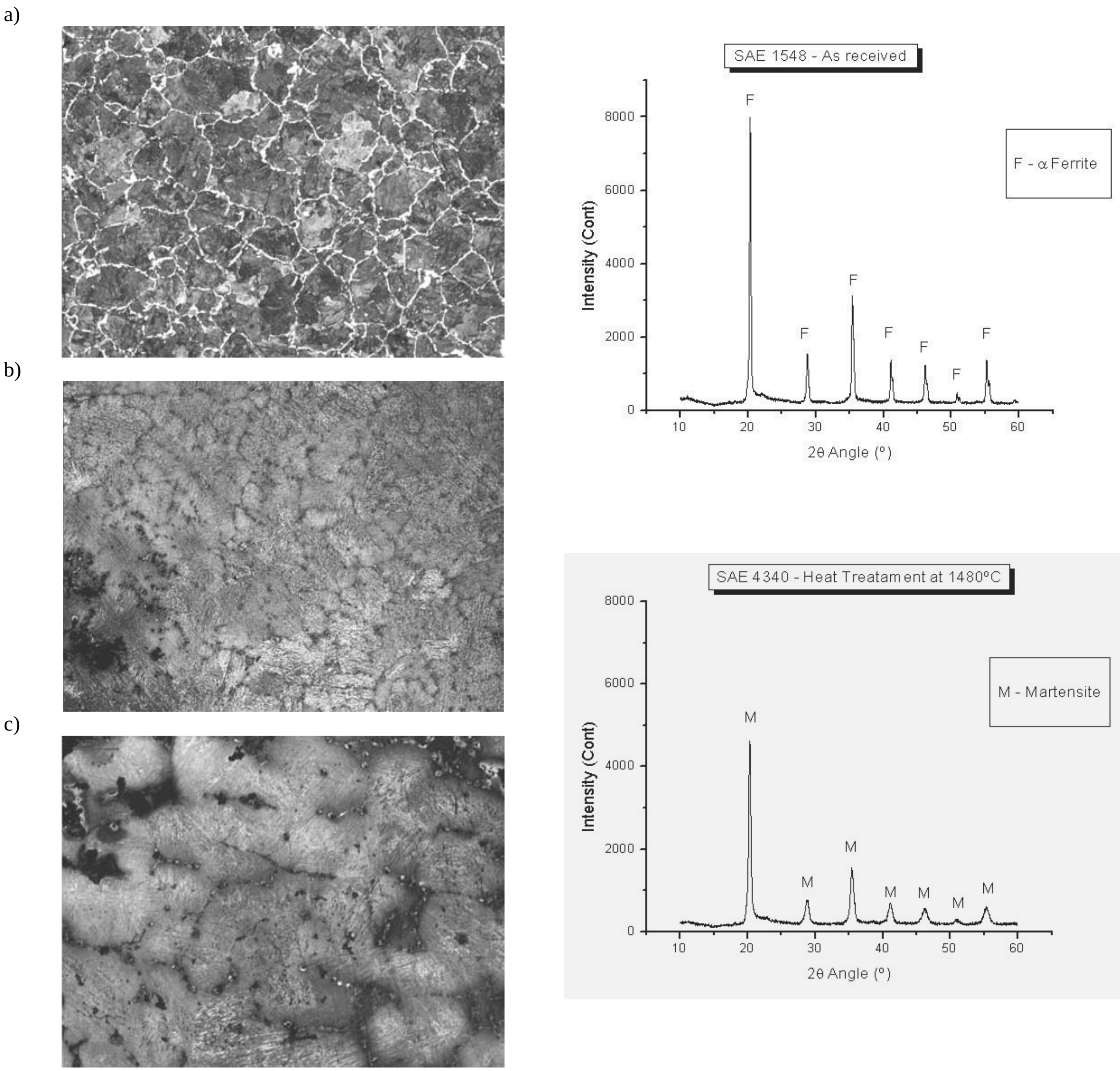


Fig. 5. Microstructures of SAE 1548 steel (optical microscopy) and correspondent XRD patterns, in different conditions: (a) as-cast; (b) after heat treatment at 1480ºC, 0 min; (c) after heat treatment at 1480ºC, 20 min. In (b) and (c) cases samples were quenched in water

SAE 1548

Typical microstructures of SAE 1548 steel produced by heating the material up to 1480ºC, holding for 0 and 20 min and then quenching in water, are shown in Figure 5; together with the original starting structure (as-cast condition). Corresponding RX diffraction patterns of each situation are also presented. SAE 1548 steel presents also hypoeutectoid composition, showing a typical pearlite + ferrite constitution in the as-cast condition, as seen in Figure 5 (a). RX diffraction pattern show only the presence of α ferrite; cementite was not detected, as its content is little – around 6% in equilibrium conditions. Dimensions of pearlite grains are in the order of 200 μm. Structures produced by heating at 1480ºC show globular morphology in the main phase, characteristic of typical thixotropic material. Globular phase also present dimensions compatible with dimensions of initial pearlite grains, showing that in the semi-solid region each austenite grain become an individual globule in the semi-solid. RX diffraction tests show the presence of only martensite in the thixotropic material.

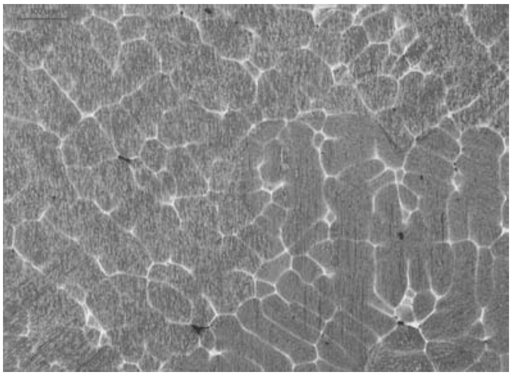
Austenite grains originated from pearlite become globular within the liquid in a very short time; however, it can be observed in Figure 5 (c) the deleterious effect of holding time on the size of primary phase globules. Interglobular regions, formed by rapid cooling of the liquid (rich in alloying elements) present in the semisolid material, show at room temperature MnFe and MnMoFe rich precipitates.

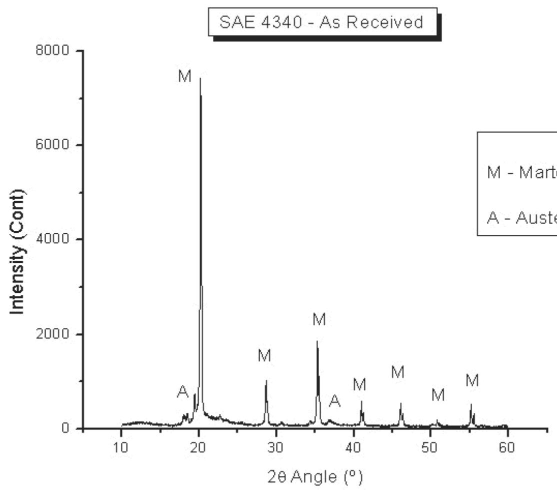
SAE 4340

Figure 6 presents microstructures of SAE 4340 steel in the forged condition and produced by partial melting at 1480ºC followed by 0min and 20min holding at this temperature. RX diffraction patterns of each microstructure are also presented in the same Figure. Due to the presence of the austenite stabilizers Cr and Ni in its composition, the structure of SAE 4340 presents essentially martensite at room temperature; spheroidal Fe, Ni, Cr, Mo and Mn carbides are also present. Figure 6 (a) shows fine martensite throughout the section; RX diffraction pattern shows the presence of some austenite as well.

Structures produced by heating at 1480ºC show also the main phase with globular morphology, typical of thixotropic semi-solid materials. RX diffraction tests indicate the presence of only martensite; probably the only phase present in the semi-solid range was austenite, meaning that the treatment temperature lays within the $\gamma + L$ region.

a)





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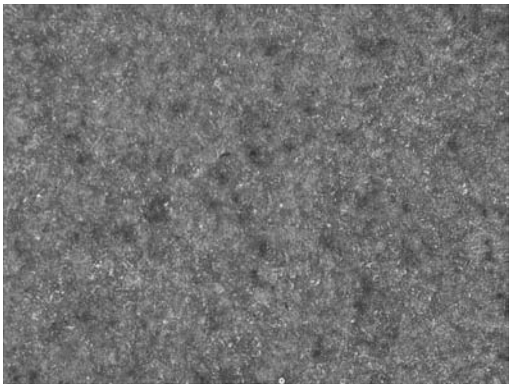

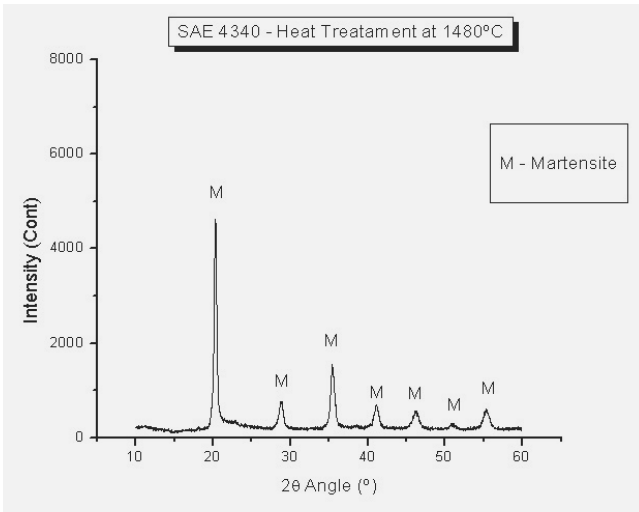

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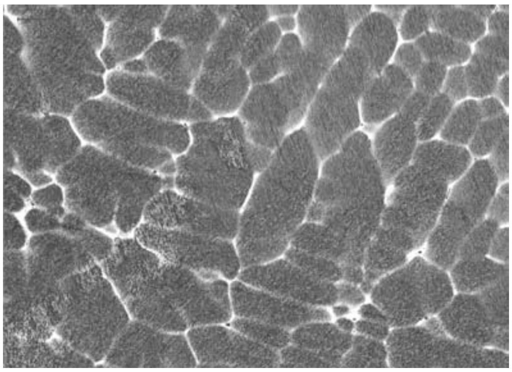

Fig. 6. Microstructures of SAE 4340 steel (optical microscopy) and correspondent XRD pattern, in different conditions: a) forged, b) after heat treatment at 1480ºC, 0 min, c) after heat treatment at 1480ºC, 20 min. In (b) and (c) cases samples were quenched in water

Carbides present in the as-cast structure can be dissolved at the high temperatures imposed in the partial melting treatment; their content in the thixocast material depends on the temperature, decreasing as temperature and holding increases. However, even at the highest investigated temperature (1485ºC) still some few surviving carbides can be observed in the interior of the globular phase of the thixocast material. Interglobular region in the structure treated at the highest temperature and higher holding time presents, according to EDS analysis, high content of Cr, Mn, Mo, Ni in solution, without any apparent precipitates.

Martensite globules in the thixocast semi-solid are fine, but coarser than the original grains, indicating the fast kinetics of globularization/growth of the solid in the liquid environment in the semi-solid region. As holding time increases, size of globular phase increases. Thixotropic material presenting fine structure can be achieved by short heating cycle to the semi-solid state – fast re-heating to $T>T_s$ and short or nil holding times; fine starting material also shall be used. Another possible technique is promoting previous deformation in the material, so recrystallization during re-heating result in a fine structure in the semi-solid. Some results from literature can bring additional information on the influence of previous deformation on austenite transformation and final grain size in ferrous alloys [14]. Moreover, to make it possible the prediction of heating conditions effects on structure transformations when producing a thixotropic semi-solid, the applicability of simulation methods developed for conventional heat treatment of steels [15] could be tested for the partial melting required to produce thixotropic material.

4. Conclusions

The work shows that the three steels investigated, SAE 1070, SAE 1548 and SAE 4340 present considerable potential for thixoforming, given by wide solidification range associated with affordable sensitivity of liquid fraction with temperature within solidification range. Although the first feature may represent a problem as far as casting is concerning due to susceptibility to high segregation and hot cracking during solidification, the second feature indicates high thixoability. SAE 1070 presented the highest thixoability, followed by SAE 1548 and SAE 4340, in this sequence. Thixoforming windows are around 50ºC for SAE 4340, 70ºC for SAE 1548 and 90ºC for SAE1070. Semi-solid produced by partial melting at the investigated temperatures is constituted in all cases by liquid and globular austenite phase. Kinetics of solid spheroidization is high in the semi-solid state, leading to globular structures in short times; increasing holding time can result in excessive growth of globular solid phase.

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