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Microstructural modifications during thixocasting AISI 304 stainless steel

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This work analyses microstructural transformations of AISI 304 stainless steel during heating to temperatures above solidus in order to produce thixocast slurries and during cooling of the semi-solid to room temperature. Samples treated at different treatment temperatures, ranging from 1300 to 1425°C were analyzed to understand the structure evolution. Results show that thixocast material can be easily produced by partial melting, resulting in a semi-solid formed by ferrite and liquid. Thixocast material presents, at room temperature, a complex structure formed by globules containing ferrite veins and austenite plates, and peritectic austenite and also some eutectic ferrite in intergranular regions.

1. INTRODUCTION

Semi-solid technology (SSM) has been widely developed for light alloys such as Al and Mg alloys. However, the advantages of the application of this technology also to high melting point alloys is bringing these materials into the SSM scenario. Some works have already reported the production of semi-solid rheo and thixocast slurries of ferrous alloys, mainly high alloyed steels[1,2]. Stainless steel strip rheocasting[3] and components made by thixocasting of high C steels[4] are examples of semi-solid processes under investigation. However, little attention has been paid to the microstructure of ferrous alloys in the rheocast or thixocast conditions. Phase transformations in the solid state can make more difficult the control of the structure during thixoforming processes, in which the semi-solid must be rapid cooled in order to prevent loss of fluidity by degeneration of the globular structure. Non-expected structures can be produced. Therefore, the advantages of the semi-solid forming could be shadowed by the possibility of producing final products with undesirable structure and properties. This can be of particular importance in case of austenitic stainless steels, which final structure when cooled from solid/liquid range is strongly dependent on cooling rate and could result in products with poor corrosion properties. This work investigates microstructural transformations during thixocasting AISI 304 steel.

2. EXPERIMENTAL PROCEDURES

Cylindrical samples (ϕ12 x 8 mm) of AISI304 steel in rolled + annealed condition were heated at temperatures from 1300 to 1425°C (from 10 to 10°C) under argon atmosphere, and water quenched. Microstructures were analyzed by optical and electronic microscopy and Xray diffraction. Electrolytic etching using NaOH solution and potassium metabisulphite etching were used to identify ferrite and austenite respectively. Ferrite content was also determined by magnetic measurements.

3. RESULTS

Figure 1 shows some representative microstructures produced during heating AISI 304 steel to the solidification range to produce thixocast slurries. The initial microstructure shown in Figure 1(a), is monophasic and presents only polygonal austenitic grains. At 1320°C the microstructure showed some formation of δ-ferrite in boundaries and triple junctions of the austenitic grains; at 1330°C the ferrite phase, with polygonal morphology, is clearly observed, as shown in Figure 1(b), at higher magnification. As temperature increases, ferrite content in the microstructure also increases, until temperature of the order of 1390°C is reached; samples quenched from higher temperatures show decrease in the ferrite content.

Values of ferrite content, measured by magnetic counting, in samples quenched from different temperatures, are presented in Figure 2. Observations of the produced microstructures showed that when higher ferrite content (at high temperatures) is present, when quenching this ferrite can no longer be stable and suffers a Widmanstatten transformation to austenite plates. Therefore, at room temperature, the structure present grains of austenite and grains containing plates of austenite and remaining veins of ferrite. This ferrite-austenite transformation decreases the total content of ferrite in the structure.

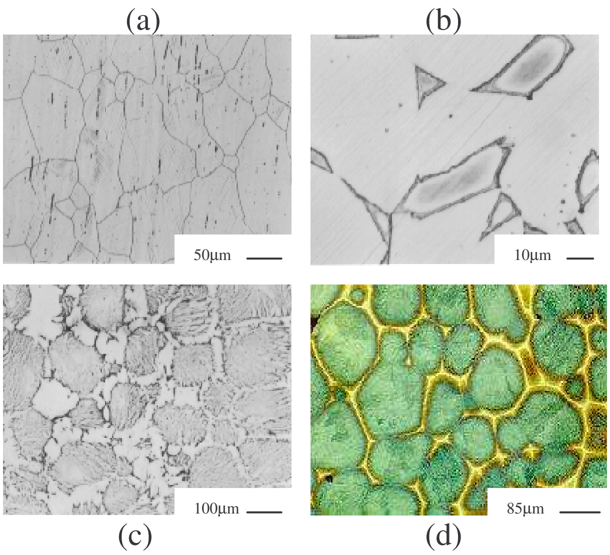


Figure 1. Microstructures of AISI304: (a) rolled + annealed condition; (b) quenched from 1330°C; (c) from 1415°C; (d) from 1425°C.

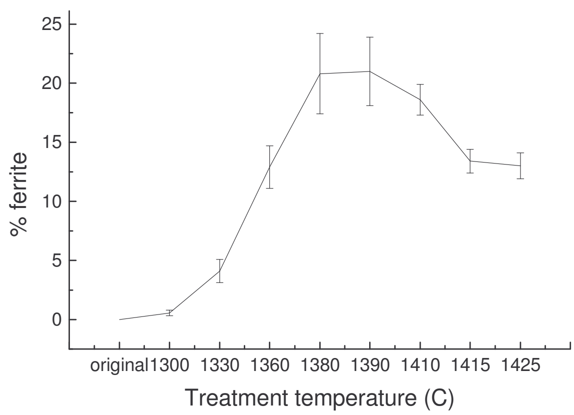
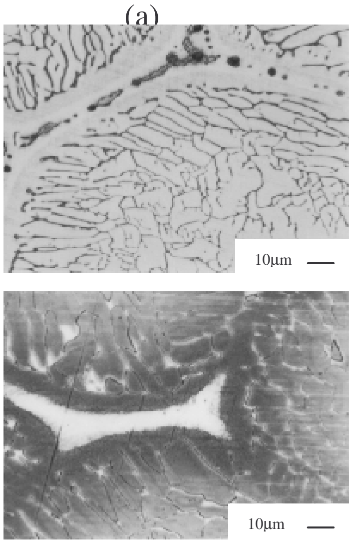


Figure 2. δ-ferrite content in AISI 304 quenched from different temperatures

At 1405°C the structure started to show liquid formation in the austenite boundaries, the presence of liquid stimulates globularisation of the ferrite and austenite grains, moved by the need to reduce superficial energy. Figure 1(c) shows microstructure cooled from 1415°C. It can be observed roundish austenite areas (lighter color) and globular grains containing austenite plates and δ-ferrite veins. These globular bi-phase grains were fully ferrite at the treatment temperature and suffered massive transformation during the rapid cooling. Thin austenite side plates grow from the interface between the austenite and δ-ferrite towards the interior of the ferrite. As temperature increases, austenite content decreases as it melts and, at 1425°C the material is semisolid, containing only ferrite grains and liquid. Ferrite grains are globular which characterizes a thixocast slurry. At room temperature, the microstructure presents, as observed in Figure 1(d), typical thixocast condition: globular primary phase (here transformed in austenite + ferrite due to the rapid cooling), surrounded by a second phase of low melting point, formed direct from liquid during cooling.

Figure 3 shows in more detail and with two different etchings, the thixocast structure produced by partial melting at 1425°C. In Figure 3(a) dark phases are ferrite (under NaOH electrolytic etching) and in Figure 3(b) dark phases are austenite (under potassium metabisulphite etching). It can be observed that the globular grains observed in the thixocast material are actually formed by plates of austenite and veins of δ-ferrite. Interglobular region presents austenite (with clear segregation from the surface of the globules to the center of the boundary), and also carbides and secondary ferrite in the center of the boundary. A presence of a compact layer of austenite can be observed around the globular primary phase. Therefore, the thixocast steel presents, in the semi-solid state, ferrite globules and liquid and, as a result of the rapid cooling, a complex structure can result. The thixocast structure produced at 1425°C is, at room temperature, no longer fully austenitic but contains around 12% ferrite.

(a)



(b)

Figure 3. Microstructure of thixocast AISI 304 steel.

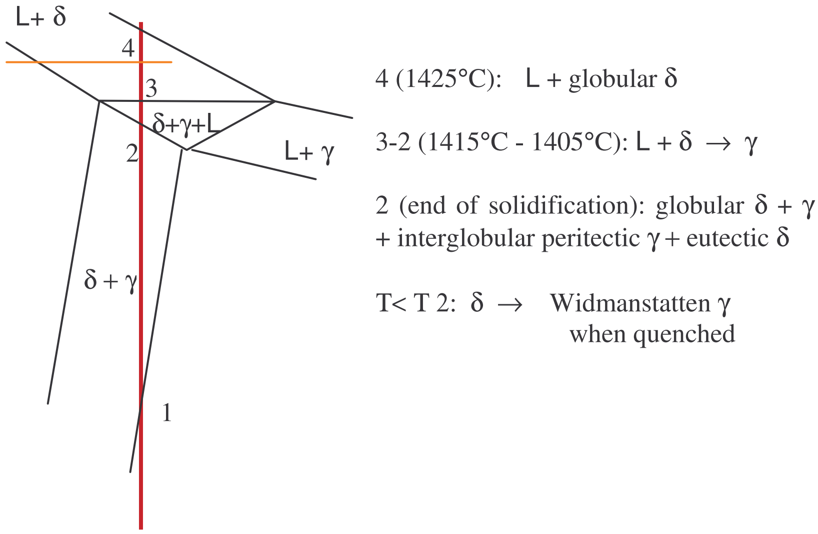


Figure 4. Probable solidification sequence for thixocast AISI 304 steel.

Considering the composition of the steel investigated and the observed microstructure transformations during heating to thixocast temperatures and cooling the slurry to room temperature, it is possible to suggest the probable phase diagram and solidification mode, presented in Figure 4. With Creq./Nieq. ~2 its solidification mode can be either F (ferrite) or FA (ferrite-austenite). The observed presence of austenite around ferrite globules indicates that FA mode is present in this case. Primary phase is ferrite at the thixocast temperature (T>1415°C), as globules immersed in liquid enriched in austenite former elements. During cooling, austenite is formed by peritectic reaction taking place in the solid-liquid interface, resulting in a layer around ferrite grains. The growth of the peritectic austenite promotes segregation of ferrite former elements to the remaining liquid. At final stages of the solidification, precipitates and ferrite can be nucleated in the enriched liquid in front of the growing peritectic austenite. As at this point the liquid is forming both austenite and ferrite, a eutectic reaction is taking place.

Even treatments at 1200°C for 30min were not sufficient to eliminate completely ferrite and precipitates, as observed in Figure 5.

Therefore, the solidified structure presents the observed globular grains of primary ferrite (not totally consumed by the peritectic reaction), peritectic austenite, eutectic ferrite and carbides. As cooling proceeds, the δ-ferrite is decomposed in thin austenite side plates. Therefore, the final thixocast product can present a very complex structure and as, observed elsewhere[1], with poor corrosion properties. Post heat treatments were investigates to promote austenitization of the thixocast structure.

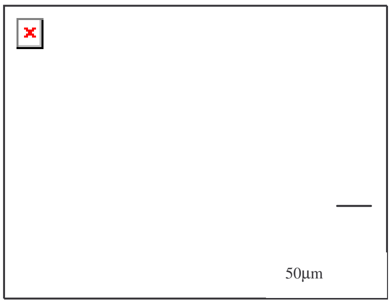


Figure 5. Thixocast AISI304 after austenitizing at 1200°C/ 90min

4. CONCLUSIONS

Results show that thixocast slurries of AISI 304 steel can be easily produced by partial melting, with the slurry presenting globular ferrite and liquid. However, the thixocast product presents a complex structure, with globular grains of Widmanstatten austenite + remaining veins of primary ferrite, peritectic austenite and eutectic ferrite, and Mo, Cr, Mn rich precipitates, due to rapid cooling usually required in thixoforming processes. Austenitization treatments are unable to recover the fully austenitic condition.

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