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CHARACTERISATION OF INTERMETALLIC INCLUSIONS OBTAINED BY DISSOLUTION OF IRON ALLOYING ADDITIVES IN MOLTEN ALUMINIUM

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Abstract

Iron is an important and cost-effective alloying additive in aluminium alloys. However, for achieving high quality and rapid alloying of iron in the aluminium industry, particularly in state-of-the-art melting systems equipped with an electromagnetic pump (EMP), a fundamental understanding of the mechanism of dissolution of iron additives is essential.

Commercial iron powder (AHC 100.29, supplier Höganäs AB, Sweden) and standard aluminium alloy AA 1050 in the form of 6mm strip (producer: Impol d.d., Slovenia) were used in this research. Pure iron powder was uniaxially compacted into mini-briquettes with a green density of about 60 % of T.D. and then cold isostatically pressed (CIP-ped) to 80 % of theoretical density (T.D). Briquettes were placed onto AA 1050 aluminium strip 6 mm thick, at the bottom of a graphite crucible. Infiltration was performed in air, at 750 ºC for 10 min.

To study the behaviour of discrete particle additions, in a separate set of experiments the original iron powder used in making tablets was mechanically stirred by rotating a graphite impeller near the bottom of the melt for 10 min. at 750 ºC. After that, all specimens were naturally cooled to room temperature, cut and polished for SEM and EDS investigation.

The results confirmed that dissolution of the initial iron particles combined with spontaneous pressureless infiltration of molten aluminium into porous iron briquettes occurs by the formation of complex intermetallic inclusions consisting of an iron-rich intermetallic core and aluminium-rich intermetallic cladding. Depending on the density of the iron briquettes, the obtained cladding is porous, consisting of individual aggregates (this is the case for low-density briquettes fabricated by uniaxial pressing), or in the form of a dense multi-layered surface coating in the case of pressed and CIP-ped briquettes. The core of low-density briquettes is completely infiltrated, while the

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core of high-density briquettes remains non-infiltrated. Individual intermetallic aggregates are well dispersed in the melt and, after solidification in the aluminium matrix, thus influencing the characteristics of the final products.

Key words: aluminium, dissolution of iron, intermetallic inclusions, SEM, morphology

Introduction

Iron is generally considered to be an undesired impurity in aluminium. However, low concentrations of iron (0.15-1.8 wt.%) in aluminium improve its mechanical properties and make it easier to roll thin aluminium sheets. Aluminium with increased iron content can also be applied in profiles, since iron improves the extrusion properties reducing the grain size. Alloys with iron and manganese near the ternary eutectic content, such as 8006, can have useful combinations of strength and ductility at room temperature and retain strength at elevated temperatures. These properties are due to the fine grain size that is stabilized by the finely dispersed iron-rich second phase. Iron is also added to the aluminium-copper-nickel group of alloys to increase strength at elevated temperatures [1].

The solubility of iron in molten aluminium is high and it is therefore easily dissolved at all molten stages of production. According to the most reliable data [2], the eutectic reaction occurs at 652-655 ºC for a melt concentration of 1.8 wt.% **Fe** at the eutectic point. In contrast, the solubility of iron in the solid state is very low (~0.05 wt.%) [2]. Therefore, most of the iron present in aluminium over this amount appears as an intermetallic second phases in combination with aluminium and often other elements. The phase usually denoted is Al_3Fe with an extensive homogeneity region, from 37.3 to 40.7 wt.% Fe. It can be described by the different formulas Al_7Fe_2, $Al_{13}Fe_4$, $Al_{19}Fe_6$, and Al_3Fe.

In addition, Al_5Fe_2, Al_6Fe, Al_5Fe, Al_9Fe_2 and others were also reported in systems proceeded with various cooling rates [2].

In the manufacture of iron-containing aluminium alloys, the addition of iron can be made in the form of iron scrap or lumps of an Al-Fe master alloy containing about 5-30 wt.% of iron. Iron powder, flakes-produced by roll compaction of iron powder, and iron powder-based tablets (briquettes or mini-briquettes) are also used because of the advantages they offer in the form of shorter dissolution time. Rapid alloying of iron is critical to maximising the efficiency of aluminium casting house operations, and is becoming particularly important in modern continuous casting operations dedicated to providing high productivity.

In order to achieve appropriate and rapid alloying of iron in the aluminium industry, particularly in state-of-the-art melting systems equipped with an electromagnetic pump (EMP), an understanding of the mechanism of the dissolution of iron additives is an emerging prerequisite.

Incomplete and non-uniform iron dissolution could seriously affect the major properties of the final product (e.g. the quality of thin gauge foils made of continuous cast aluminium strip of allying series 8xxx). Moreover, it could also disturb pumping of the liquid metal through the EMP, causing partial or even complete blockage of the ceramic tube inside the EMP pump. Note that the main reason for such a costly blockage is deposition of iron-rich aggregates, originating from incomplete and non-uniform iron dissolution, on the surface of the ceramic tube inside the pump, the connecting pipe and the returning leg. Once incompletely dissolved, the iron containing particulates will be additionally stimulated by the alternating magnetic field of the EMP for coagulation into larger aggregates [3].

Therefore, due to the increasing interest of the aluminium industry for introducing rapid melting technologies, but still limited technical information on the dissolution of iron additives in molten aluminium, a more detailed investigation of this topic is requested.

Hence, in the present study, the morphology and chemical composition of various intermetallic inclusions obtained by performing laboratory scale dissolution of iron particles and pressed tablets (mini-briquettes) in molten aluminium were investigated by scanning electron microscope (SEM). The purpose of this investigation was to identify the mechanism of iron dissolution in molten aluminium and to correlate the morphology of the obtained inclusions, which are influencing the quality of the molten metal and solidified end products, with the initial morphology of the applied iron particles.

Experimental work

Commercial iron powder (AHC 100.29, supplier Höganäs AB, Sweden) with an average particle size of 70 µm with no particles larger than 200 µm and standard aluminium alloy AA 1050 in the form of 6mm strip (producer: Impol d.o.o., Slovenia) were used for this research. Pure iron powder was compacted without addition of any binder phase into mini-briquettes (Ø =10 mm, H=20 mm). Two grades of these mini-briquettes were made. Low-density briquettes with a green density of about 60% theoretical density (T.D.) were uniaxially compacted at 100 MPa, whereas high-density briquettes with a green density of about 80% T.D. were made by combining uniaxial pressing under 100 MPa and cold isostatic pressing (CIP-ing) under 300 MPa.

The green density of mini-briquettes was calculated by measuring the dimensions of the compacted samples and weight.

Mini-briquettes were placed onto AA 1050 aluminium strip 6 mm thick, at the bottom of a graphite crucible. The volume of aluminium strip was selected to exceed the volume of pores in the mini briquettes by 50 vol.%, in this way providing sufficient liquid infiltrant for complete infiltration of the porous iron briquettes.

Infiltration was performed in air, at 750 ºC for 10 min.

To study the behaviour of discrete particle additions in a separate set of experiments, the original iron powder used in making the mini-briquettes was mechanically stirred, by rotating a graphite impeller near the bottom of the melt for 10 min. at 750 ºC.

After that, all specimens were naturally cooled to room temperature, cut and appropriately polished for SEM-EDS investigation.

Results and discussion

An immediate visual inspection of cooled samples showed a significant dissolution of the initial iron mini-briquettes (both pressed and pressed + CIP-ped) in molten aluminium.

SEM investigation of the non-dissolved part of the low-density iron mini-briquettes confirmed that after 10 min. at 750 ºC full infiltration had been achieved (Figure 1).

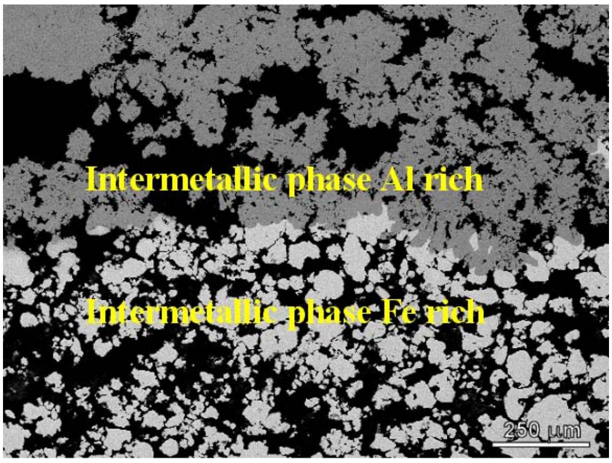

Figure 1. SEM micrograph of the cross section of an iron powder briquette after submersion for 10 min. in molten aluminium. The microstructure obtained is porous, with a well-emphasized interface between the iron-rich core and the aluminium-rich cladding. Phase compositions determined by EDS correspond to $Al_{0.2}Fe_{0.8}$ *for the core and* $Al_{0.7}Fe_{0.3}$ *for the cladding.*

The obtained microstructure is porous, with a well-emphasized interface between the iron-rich core and the aluminium-rich cladding. Phase compositions determined by EDS correspond to $Al_{0.2}Fe_{0.8}$ for the core and $Al_{0.7}Fe_{0.3}$ for the cladding. In addition, a more detailed investigation of the interface region showed the presence of a thick intermediate $Al_{0.45}Fe_{0.55}$ third phase, placed in between the core and the cladding (Figure 2). The intermediate $Al_{0.45}Fe_{0.55}$ phase was found to be porous (Figure 3), in contrast to $Al_{0.2}Fe_{0.8}$ and $Al_{0.7}Fe_{0.3}$, consisting of fully dense aggregates (Figure 2) in a porous body (Figure 1).

There was, however, a significant difference in the average particle size of these aggregates. The individual $Al_{0.7}Fe_{0.3}$ aggregates are larger than $Al_{0.2}Fe_{0.8}$, as evident in Figure 1, which is most probably caused by further coarsening assisted by infiltration.

A possible explanation for the formation of a characteristic core-cladding structure is that simultaneously with the propagation of the infiltration front, the counter-current motion of the diffusion front, caused by dissolution of iron in molten aluminium, also proceed, leading to the formation of the core-cladding interface. The result is the graded multi-layered structure of the infiltrated part of the specimen, presented in Figure 1.

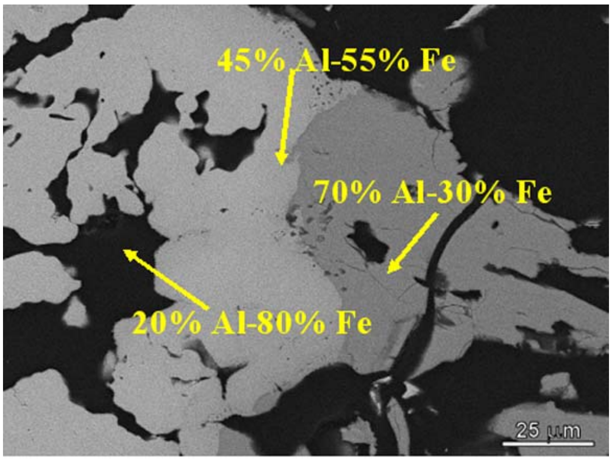

Figure 2. Various Fe-Al intermetallic phases obtained by simultaneous dissolution of iron in molten aluminium combined with infiltration of molten aluminium into a low-density (pressed) briquette.

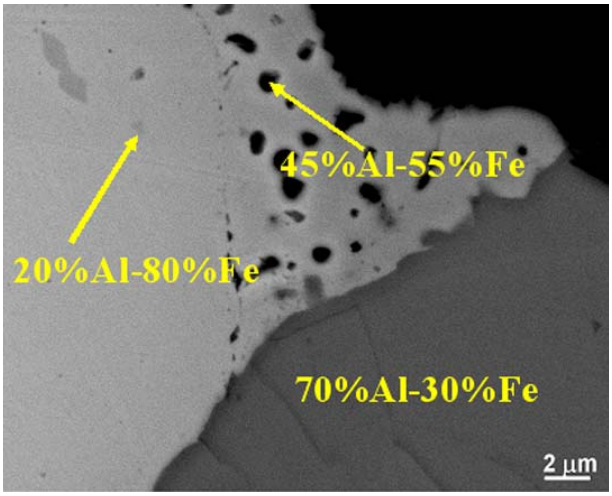

Figure 3. SEM micrograph of the reaction front revealing the presence of a thick intermediary porous layer consisting of $Al_{0,45}Fe_{0,55}$.

Propagation of the infiltration front occurred across several teeth-like infiltrating areas, growing from $Al_{0.7}Fe_{0.3}$ into $Al_{0.2}Fe_{0.8}$ (Figure 4).

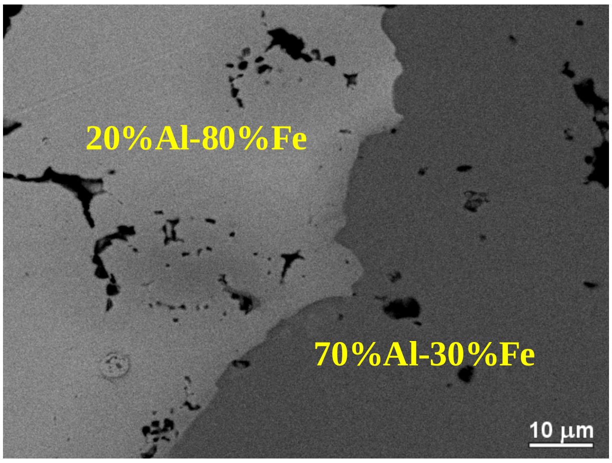

Figure 4. SEM micrograph of the solidification front of an aluminium-rich phase showing many teeth-like infiltration areas, growing from $Al_{0.7}Fe_{0.3}$ *to* $Al_{0.2}Fe_{0.8}$.

In contrast to the completely infiltrated low-density iron mini-briquettes, infiltration of the high-density iron mini-briquettes was limited just to the surface region (Figure 5).

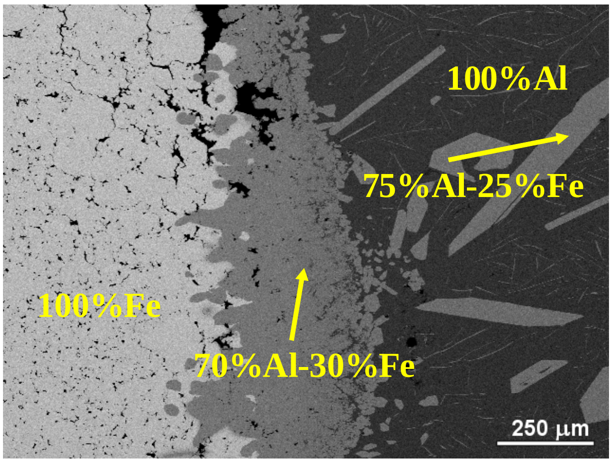

Figure 5. SEM micrograph of the characteristic core-cladding structure obtained by simultaneous infiltration and dissolution processes in a high-density (pressed+CIP-ped) briquette during its submersion in molten aluminium.

Behind the aluminium infiltration-iron dissolution front, approximately 300 μm thick cladding with exactly the same composition ($Fe_{0.8}Al_{0.2}$ and $Al_{0.7}Fe_{0.3}$) as in the previous set of experiments performed with pressed mini-briquettes was observed in Figure 6. The only exception was the intermediate $Al_{0.45}Fe_{0.55}$ phase, which here was not detected. The non-infiltrated iron core in the middle and the solidified aluminium on the surface of the high-density mini-briquette, with Al_3Fe needle-like inclusions created by dissolution of iron in molten aluminium, are two other important regions observed in the microstructure in Figure 5.

Instead of the porous packed individual inter-metallic aggregates obtained by infiltration of low-density iron mini-briquettes, almost fully dense infiltrated cladding in high-density iron mini-briquettes was achieved (Figure 6).

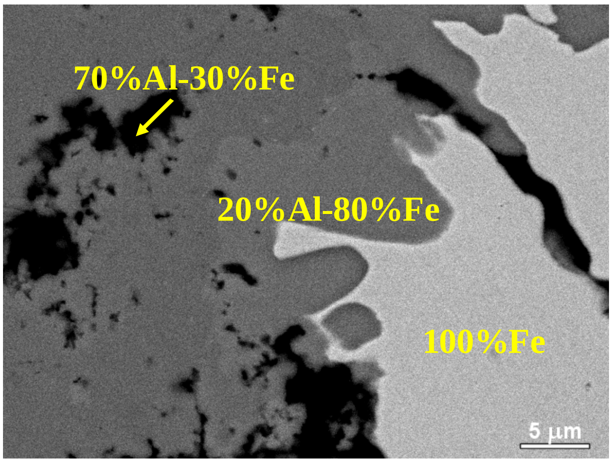

Figure 6. SEM micrograph of the cladding area in a high-density briquette.

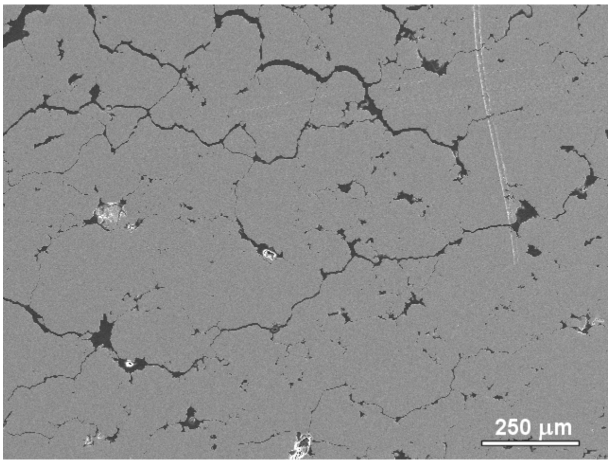

Figure 7. SEM micrograph of a high-density iron briquette revealing densely packed individual iron particles.

SEM investigation of the microstructure of the solidified aluminium on the surface of infiltrated samples showed the presence of several large crystals and many fine particles (with the same composition $Al_{0.75}Fe_{0.25}$ corresponding to $FeAl_3$), dispersed in a pure aluminium matrix. This finding additionally proves a significant amount of iron diffusion from mini-briquettes to molten aluminium (Figures 7-9).

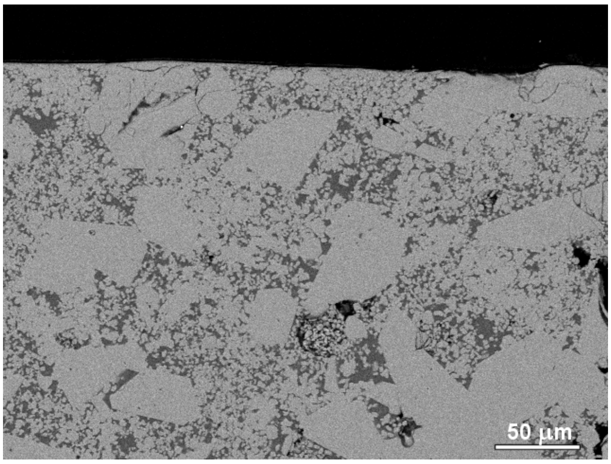

Figure 8. SEM micrograph of the aluminium phase solidified at the surface of a high-density briquette after submersion in molten Al showing the presence of a few large and many fine $Fe_{0,25}Al_{0,75}$ *crystals with chemical composition corresponding to* $Fe_{0,25}Al_{0,75}$ *dispersed in an aluminium matrix.*

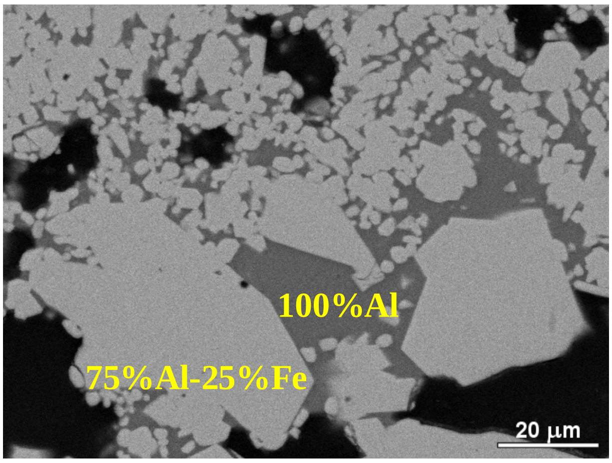

Figure 9. Detail of large and several fine $Fe_{0,25}Al_{0,75}$ *crystals dispersed in an aluminium matrix.*

In the case of individual iron particles dispersed in the melt, they evidently react with molten aluminium, transforming into intermetallic aggregates with a compact $Al_{0.2}Fe_{0.8}$ core and porous $Al_{0.75}Fe_{0.25}$ cladding ((Figure 10).

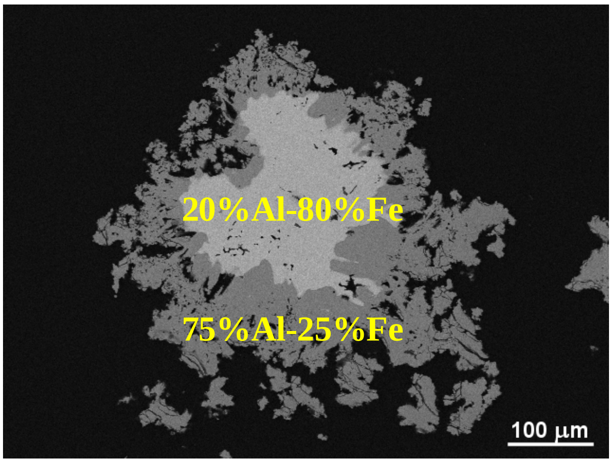

Figure 10. Core-cladding structure of an individual iron particle after submersion in molten aluminium. Elemental iron is absent from the obtained intermetallic aggregate whose size corresponds to the size of the initial iron particle.

However, it is important to note that, although after 10 min. at 750 ºC in molten aluminium the initial iron particles are completely chemically transformed, the size of the resulting intermetallic aggregate remains the same, thus affecting the performance characteristics of the final product.

Conclusion

The counter-current diffusion of iron and aluminium atoms is crucial for dissolution of individual iron particles as well as compacted iron mini-briquettes in molten aluminium. As confirmed by SEM investigation, dissolution of initial iron particles proceeds through the formation of complex intermetallic inclusions consisting of an iron-rich intermetallic core and an aluminium-rich intermetallic cladding. Inclusions are well dispersed in the molten metal, as well as (after solidification) in the aluminium matrix, thus influencing the characteristics of the final products.

The preliminary results obtained in this study indicate that the size and morphology of these inclusions do not necessarily correspond to the morphology and size of the initial iron particles. However, many inclusions were found to be even larger than the initial iron particles. This is particularly favoured by using iron mini-briquettes instead of discrete powder. Submerging iron mini-briquettes in liquid aluminium results in the formation of large aggregates consisting of: (i) a core of non-reacted compacted iron particles, (ii) cladding, fully converted into a layer of various intermetallic particles, and (iii) a boundary layer of molten metal with some individual intermetallic crystals. Because these complex aggregates cannot be dispersed by further dissolution, they strongly influenced the quality of the molten metal and the solidified end products.

Acknowledgement

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