



# Master alloys for liquid phase sintering: Some key points for the design

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The master alloy (MA) concept was brought up in the early 1970s as a means to introduce alloying elements with high oxygen affinity in low alloy sintered steels. Afterwards, it evolved to become what we currently understand: a MA as a powder that – admixed in very small amounts – can be used as a vehicle to efficiently tailor the microstructure and final properties of the steel through alloying effects. One of the main advantages of the MA approach is the unique opportunity of designing the alloy composition to enhance sintering through the formation of a liquid phase with tailored characteristics. This work presents an overview on some critical aspects to consider during the design of a MA composition. Particular emphasis is devoted to the interactions between MA particles and iron base powders which divides the design possibilities into two main families: dissolutive (non-infiltrating) and non-dissolutive (infiltrating) liquids, with significant differences in the final microstructures and dimensional stability achieved in the sintered parts. Moreover, when advanced alloying systems containing Cr, Mn and Si are considered, the study of the interaction with the base powder must also include the chemical reactions occurring during sintering. In this latter case, application of high purity sintering atmospheres might not be enough to avoid the formation of stable oxides, since the source of oxygen is in most cases the iron base powder itself.

## Introduction

Compared to the prealloying route, the MA approach presents interesting advantages such as the possibility of preserving the compressibility of the base powder, and the flexibility in the selection of the final composition of the steel. But probably one of the most interesting benefits of using a MA is the fact that its composition can be specifically designed to promote the formation of a liquid phase that enhances the distribution of alloying elements and accelerates the sintering mechanisms.

The use of thermodynamic and kinetic software tools has triggered interesting developments in the design of liquid phases, by allowing a systematic search of low melting point compositions in a targeted alloying system [1–3]. Although all these advances have led to potential industrial developments, the strict demands in terms of dimensional stability, microstructure or

density of the final parts must be met, which in turn requires a precise control over those properties, ideally from the design stage. Moreover, if the addition of cheaper and more efficient alloying elements such as Cr, Mn and Si is considered, their higher sensitivity to oxidation will require a more demanding control of the sintering atmosphere [4].

The interest in predicting and controlling the final properties of steels manufactured through the MA route has motivated the last progresses in the MA approach which aim not only to care about the composition and thermal properties of the MA powder itself, but also to consider the chemical reactions and to tailor the properties of the liquid and its behavior in contact with Fe base powders. The most recent advances performed with Fe–Mn–Si [5,6] and Cu–Ni–Si systems [7] go into this direction and resulted in a design methodology that predicts the features and evolution of the liquid during the early stages of sintering: wettability, infiltration ability and dissolutive character [8].

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This paper summarizes some of the most relevant results obtained by the authors when working with master alloys of different compositions: Cu-based, Ni-based and Fe–Mn–Si systems. The attention focuses on how the chemical reactions occurring during sintering – due to the interaction between solid, liquid and gaseous phases – affect the final properties of the steel, particularly, its dimensional behavior and microstructure formation. The objective is to be able to directly adapt the alloy design (before MA powders are atomized) to the final requirements of the sintered steel.

## Experimental procedure

The design of the MA compositions was carried out following a methodology that allows studying the liquid properties before producing the MA powder. Their characteristics were analyzed using a combination of thermodynamic software tools, macroscopic wetting/infiltration experiments and advanced thermoanalytical techniques [6,9].

This paper presents a comparison of four different liquid phases: plain Cu and three different MAs (contents in mass%): MA1 (Cu–2Ni–1Si), MA2 (Ni–4Cu–12Si) and MA3 (Fe–40Mn–15Si–1C) whose characteristics are presented in Table 2. The properties of steels modified with 4 wt.% additions of Cu, MA1, MA2 or MA3 powders have been studied to describe the chemical reactions during sintering, as well as their microstructural evolution and dimensional behavior.

A summary of the equipment and the characteristics of the experiments used are summarized in Table 1, together with references in which the specific experimental procedures are explained in detail.

## Results and discussion

### Infiltration properties of the liquids

The infiltration behavior of a liquid represents its ability to penetrate and spread through the pore system of a green compact with a specific chemical composition. Infiltration tests were carried out placing a small ingot with the composition of the liquid precursor on top of a green compact of Fe powder pressed at 600 MPa. One of the most significant features of the infiltration behavior of the liquids is that it is strongly determined by the solubility condition between the liquid phase and the solid substrate. This effect is clearly illustrated in Figs. 1 and 2, which shows the (pseudo)binary

TABLE 2

**Characteristics of the MA compositions used as liquid precursors.  $T_{\text{sintering}}$  represents the expected sintering temperature.  $T_{\text{solidus}}$  and  $T_{\text{liquidus}}$  were measured by DTA (in Ar).**

MA1	Cu–2Ni–1Si (wt.%), fraction < 45 $\mu\text{m}$ , water atomized $T_{\text{solidus}} = 1073\text{ }^{\circ}\text{C}$ , $T_{\text{liquidus}} = 1091\text{ }^{\circ}\text{C}$
MA2	Ni–4Cu–12Si (wt.%), fraction < 45 $\mu\text{m}$ , water atomized $T_{\text{solidus}} = 1084\text{ }^{\circ}\text{C}$ , $T_{\text{liquidus}} = 1127\text{ }^{\circ}\text{C}$
MA3	Fe–40Mn–15Si–1C (wt.%), Fraction < 45 $\mu\text{m}$ , gas atomized in $\text{N}_2$ $T_{\text{solidus}} = 1060\text{ }^{\circ}\text{C}$ , $T_{\text{liquidus}} = 1072\text{ }^{\circ}\text{C}$

phase diagrams of the different liquid phase formers (Cu, MA1, MA2 and MA3), represented with increasing wt.% Fe, as well as with the typical microstructures observed on the cross section of the samples after the infiltration experiments. Two different families can be clearly identified in these experiments:

- Non-dissolutive (infiltrating) liquids: Cu and MA1 (Fig. 1). These systems present low solubility of Fe in the liquid phase (highlighted as  $S_{\text{S} \rightarrow \text{L}}$  in the graph) and low solubility of the liquid-forming elements in the solid ( $S_{\text{L} \rightarrow \text{S}}$  in the graph). As a consequence, they provide an excellent infiltration behavior, as the low degree of interaction between the liquid and solid phase allows a rapid penetration of the liquid in the iron-skeleton.
- Dissolutive (non-infiltrating) liquids: MA2 and MA3 (Fig. 2). These two systems, in contrast, present higher solubility values ( $S_{\text{S} \rightarrow \text{L}}$  and  $S_{\text{L} \rightarrow \text{S}}$ ). In these cases the infiltration process stops shortly after the liquid formation as the dissolution of the iron substrate and subsequent re-precipitation of solid phases closes the access to the pore channels.

Here it must be stressed that all the liquids studied here are generated by spontaneous melting and not by eutectic reactions between the MA particles and the base metal (see [10]).

### Wetting characteristics of the liquids on Fe and Fe–C substrates

The ability of the different liquids to wet and spread over an iron surface was evaluated using macroscopic wetting angle measurements (Fig. 3). These experiments were carried out in two different atmospheres (inert Ar or slightly reducing  $\text{N}_2$ –10% $\text{H}_2$ ) and using two different types of substrates, plain Fe or steel substrates with a composition Fe–0.5 wt.%C.

The presence of reducing agents either in the atmosphere ( $\text{H}_2$ ) or in the substrate (C in Fe–0.5C substrate) significantly improves

TABLE 1

**Summary of the different experimental techniques, equipment and characteristics of the experiments.**

Technique	Equipment and characteristics of the experiment	Ref.
Thermodynamic calculations	ThermoCalc <sup>®</sup> software. Databases: TCFE5 for Fe based MAs, and SSOL4 for Ni and Cu based MAs.	[6–9]
Wetting/infiltration experiments	DSAHT furnace (KRÜSS) equipped with an observation window and a recording system. Atmospheres: Ar and $\text{N}_2$ –5% $\text{H}_2$ (purity 99.9%). Videos recorded during continuous heating at 5 $^{\circ}$ /min heating rate up to 1200 $^{\circ}\text{C}$ .	[5–9]
STA: Differential thermal analysis and thermogravimetry	Netsch STA 449 C	[14–17]
Dilatometry	Experiments in Ar atmosphere. 1500 $^{\circ}\text{C}$ , heating/cooling rate 20 $^{\circ}$ /min Netsch 402 C dilatometer	
Mass spectrometry	Experiments in Ar, $\text{H}_2$ atmosphere. 1300 $^{\circ}\text{C}$ , heating rate 10 $^{\circ}$ /min Quadrupol mass spectrometer Netzsch QMS 403 Aeölos Coupled by a quartz capillary with STA and dilatometer. Allows the analysis of the gaseous species evolved during the thermal treatment	
Step sintering experiments	DT1000 Adamel Lhomargy quenching dilatometer Atmosphere: He, Heating rate 10 $^{\circ}$ /min, Cooling rate $\sim$ 100 $^{\circ}$ /s	[6–9]

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