Preliminary Results from Duplex Procedure for Obtain of Fe Based Materials for Automotive Applications

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Abstract. Iron based materials still represent a high percentage from metallic materials used in industry, in general, and in automotive industry, in particular. In this case we used a duplex process in order to obtain the FeMnSiAl experimental alloy for a more efficient use of various units. In the first stage iron, manganese, silicon and aluminum were melted and mixed together using arc melting technology and for the second stage the alloy was re-melt for homogeneity in an induction furnace. Chemical composition, after each melting step, was analyzed using EDS Bruker detector for various areas and microstructural characterization using SEM, Vega Tescan LMH II with SE detector, equipment. This alloy is proposed as a metallic approach of mechanical dumpers used in automotive industry for low and medium impact contacts.

1. Introduction
The duplex process, which was first used in the second half of the 19th century, makes possible more efficient use of various units. For example, the duplex process using a basic open hearth furnace and an arc furnace or a converter and an arc furnace is used to increase the output of the electric furnace (that is, to increase the output of high-quality steel) and to reduce the specific consumption of electric power. This decrease is accomplished by removing from the electric furnace such operations as the melting of the charge and the partial refining of the metal; in the duplex process, only the final refining and deoxidation of the steel are carried out in the arc furnace.

The application of the duplex process is limited [1-5]. Shape memory alloys present a high damping capacity especially in the transformation domain from austenite to martensite and reverse based on dislocations movement, plates re-orientation and other phenomena already discussed [6-9]. From shape memory alloys categorizes most used is NiTi but this one have a quite expensive production procedure and cannot be suitable for automotive applications at industrial scale.

By economical point of view is more suitable to obtain a Fe-based shape memory alloy and the system FeMnSi present very good results in damping capacity as well as shape memory effect [10-13]. Aluminium element can be used as additional element to system FeMnSi in order to improve the mechanical damping capacity.

In this paper is presented few experimental results from the obtaining part of this alloy using two melting methods by arc melting and induction melting for chemical homogenization.
2. Experimental setup

An experimental alloy, FeMnSiAl, was proposed and obtained from high purity materials. Vacuum Arc Melting Facility MRF ABJ 900 (University Politehnica Bucharest) ensures the melting of metallic materials in argon-controlled atmosphere after pre-emptive working chamber up to 10-5 mbar by means of a non-consumable throttle tungsten mobile electrode. The RAV MRF ABJ 900 has the following technical features: The working chamber (vacuum container) is made of 304L stainless steel and is provided with double water cooled walls (temperature below 500°C). The base of the working chamber is made of aluminium with a thickness of 20 mm. It is placed above the electrical generator to minimize the surface and volume of the plant. The installation loading is done at the bottom of the work stand by opening the articulated bell and placing the load in the alveoli configured in the copper base plate. The loading of the materials for elaboration and refining, as well as the unloading of the samples, is done simply by lifting the lid that will be hung with a hinge from the rest of the room. Visualization of the crucible and electrode can be performed through the 4" diameter window located at the front of the plant. The work area illumination during the operation of the installation is through a 1" diameter window, placed in the rear of the container with a halogen lamp included. The viewing window is equipped with a folding welding glass to protect the view during melting and refining of the metallic load. The vacuum pump system is connected to the bottom of the working chamber. The rewinding part, made at Gheorghe Asachi Technical University in Iasi, took place in an induction furnace and the materials were heated to a temperature of 1600°C.

The chemical analysis of the materials was performed with an EDS - Bruker EDAX spot detector or by line distribution of the main elements Fe, Mn, Si and Al. The microstructural alloy was analyzed by SEM VegaTescan LMH II electronic scanning microscopy after the samples were mechanically polished with SiC metallographic maps and etching with nital solution followed by rinsing with distilled water.

3. Experimental results

The experimental alloy was first obtained using arch melting technique and secondly induction furnace for chemical homogenization. The experimental composition proposed for obtaining was Fe15Mn3Si3Al. In figure 1 a) is presented the image of the surface and the chemical analyze points proposed for analyze on the inferior part of the ingot and b) XRD spectrum of sample FeMnSiAl in three points (1, 3 and 5 from figure a)).

![Figure 1](image_url)

**Figure 1.** a) Image of the surface and the chemical analyze points and b) XRD spectrum of sample FeMnSiAl in three points (1, 3 and 5).
The material has a mixed microstructure which consists mainly of γ-austenite (111 and 200) but also some reduced peaks of ε-martensite (100), figure 1b) at room temperature.

The necessity of the second melting stage appears after we analyze the ingot on the length and we observe high variations between the ingot ends, not only for chemical composition but also for magnetic properties.

We propose a chemical composition analysis from the center of the sample, point 1 to exterior, point 5 and determine the homogeneity of the ingot on the transverse way. The XRD results, figure 1b), present no variation between the selected points (1, 2 and 3) so no chemical variation are observed on this part of the ingot. The results are presented in table 1, in wt% and at%, also with the equipment error. Small variations between the same element chemical composition and beside the average value and with some low differences for Mn, Si and Al against the proposed chemical composition (15, 3 and 3 wt%).

| Elements/points | Fe     | Mn     | Si     | Al     |
|-----------------|--------|--------|--------|--------|
| Pt. 1           | wt[%]  | at[%]  | wt[%]  | at[%]  | wt[%]  | at[%]  |
| Pt. 2           | 74.85  | 71.51  | 20.92  | 20.31  | 2.52   | 4.79   | 1.17   | 3.39   |
| Pt. 3           | 76.14  | 70.88  | 20.18  | 19.08  | 2.23   | 4.12   | 1.53   | 2.94   |
| Pt. 4           | 75.36  | 70.33  | 20.74  | 19.66  | 2.49   | 4.61   | 1.69   | 3.25   |
| Pt. 5           | 76.08  | 71.80  | 20.85  | 19.99  | 1.98   | 3.72   | 1.20   | 3.04   |
| Average         | 75.66  | 71.56  | 20.74  | 19.94  | 2.25   | 4.216  | 1.34   | 2.97   |
| Error %         | 1.5    | 0.35   | 0.15   | 0.10   |

By chemical analyze on the bottom part of the ingot we observe a higher percentage of Mn, as against top part (11.5%) so during the preparation of the materials for arch melting and the process itself appear this variation.

In table 2 are presented the results of chemical analyze made on six points. For induction melting we wire cut, the initial ingot and put all the pieces in a ceramic crucible. Melting starts around 1600°C temperature and during 30-60 seconds for homogenization, under Ar atmosphere and poured in a metallic pre-heated form. The ingot obtain was more 60% weight from the starting mass. It was lost some material from the first quantity that remain stuck on the crucible.

| Elements/points | Fe     | Mn     | Si     | Al     |
|-----------------|--------|--------|--------|--------|
| Pt. 1           | wt[%]  | at[%]  | wt[%]  | at[%]  | wt[%]  | at[%]  |
| Pt. 2           | 81.43  | 77.96  | 14.44  | 14.06  | 2.37   | 4.51   | 1.75   | 3.48   |
| Pt. 3           | 81.48  | 78.52  | 15.07  | 14.76  | 2.09   | 4.0    | 1.36   | 2.72   |
| Pt. 4           | 80.37  | 76.33  | 14.69  | 14.18  | 2.88   | 5.44   | 2.06   | 4.06   |
| Pt. 5           | 79.97  | 76.01  | 15.18  | 14.66  | 2.88   | 5.45   | 1.97   | 3.88   |
| Pt. 6           | 80.07  | 75.71  | 14.54  | 13.98  | 2.95   | 5.55   | 2.44   | 4.77   |
| Average         | 80.41  | 76.48  | 14.79  | 14.29  | 2.75   | 5.19   | 2.10   | 4.04   |
| Error %         | 1.5    | 0.35   | 0.15   | 0.10   |

In figure 2 the distribution of main elements Fe, Mn, Si and Al are presented in the selected image from 2a). The distribution of the elements is homogeneous for all the elements including Al. For alloys with shape memory effect and damping capacity is important for the alloy to present a high
chemical homogeneity, even if the damping capacity usually decrease with elimination of defects, chemical variations etc.

Figure 2. Chemical elements Fe, Mn, Si and Al after the re-melting operation a) area selected for distribution analyze, b), iron distribution, c) manganese distribution, d) silicon distribution and e) aluminium distribution.

The alloy microstructure is presented in figure 3 using SEM technology. The sample is after heat treatment of solution hardening with 0% deformation so the structure suppose to be austenite.

Figure 3. SEM images of FeMnSiAl experimental alloy a) 500x and b) 1000x.
The present of martensite plates, figure 3 b), can be put on the contraction forces that appear at cooling stage after the heat treatment at 1100°C and chill in oil. The appearance of a relief on the austenite structure was reported in CuMnAl superelastic alloy [14]. The alloy presents at room temperature a stable austenitic phase (A, γ, FCC) [15-17]. The stacking fault energy of austenite phase is an important factor in controlling the deformation behaviour and the occurrence of deformation in this kind of materials.

Fe–Mn–Si-based alloys for specific compositions are expected to have a shape memory effect associated with the deformation-induced martensitic transformation from face-centred cubic austenite to hexagonal martensite on loading.

As further work we will obtain the hot-rolled samples and analyze the damping behaviour of the materials using Dynamic Mechanical Analyzer (DMA) in order to obtain the coefficient tand values with temperature and frequency variation. Also the materials will be analyzed using an laboratory pendulum to establish the internal friction with and without a pre-strain value.

4. Conclusions
We obtain a Fe-based alloy with possible shape memory effect (based on the chemical composition) and present some preliminary results about the chemical composition evolution in the duplex melting process. At room temperature the material have a microstructure from especially from γ-austenite but also with reduced quantities of ε-martensite. The experimental sample, FeMnSiAl, after the heat treatment of solution hardening with, have no deformation degree applied, so the structure suppose to be austenite but from the scanning electron microscopy images it seems to present martensite plates that can be put on the contraction forces phenomena that appear at cooling stage.

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Acknowledgement
This work was carried out through the Partnerships in priority areas program - PN II, implemented with the support MEN - UEFISCDI, project no. PTE 48/2016.