Experiment Proposal

Experiment number GP2023017

Principal investigator	Dr Elena Villa, CNR ICMATE Unit of Lecco, ITALY
Co-investigator (*)	Miss Francesca Villa, CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	TEM investigation of NiMn-based alloys for elastocaloric and magnetocaloric properties

MRF Instrument	**High Resolution TEM**	**Days requested:**	5
Access Route	Direct Access	**Previous GP Number:**	no
Science Areas	Energy, Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-

Similar Submission? we have submitted a proposal for investigation in diffraction beam line in ESRF at Grenoble of NiMnGa samples. This proposal was accepted and performed in the september 2022

Industrial Links -

Non-Technical Abstract

Our work concerns the development of multicaloric material based on Shape Memory Alloys, with elastocaloric and magnetocaloric properties. The development of their functional properties to apply these materials in solid state refrigeration involves the tuning and the investigation of the microstructure, starting from thermomechanical approach on cast samples to preparation of sintered alloys. In recent studies we obtain interesting properties for elastocaloric NiMnGa developed by sintering process after ball milling, in which a particular microstructure is prepared with stored mechanical energy which gives new mechanical perspective for applications, maybe also in magnetic/mechanical coupling. Therefore, we propose an exhaustive registration of TEM images to catch the microstructural key features which are the origin of this complex mechanical and magnetic performances.

Publications

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ISIS neutron and muon source **IM@IT E-platform:** No

Instruments	**Days Requested:**
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Sample record sheet

Principal contact Miss Francesca Villa, CNR, ITALY
MRF Instrument **High Resolution TEM** **Days Requested:** 5
Special requirements:

SAMPLE

Material	NiMnGa	NiMnTi	-
Formula	Ni50Mn30Ga20 at%	Ni50Mn35Ti15 (at%)	-
Forms	Solid	Solid	
Volume	1 cc	1 cc	
Weight	500 mg	500 mg	
Container or substrate	none	none	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	300 - 300 K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	none	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	ULTRAMICROTOME RMC PowerTome PC with CR-X Cryosectioning system for preparation of ultrathin section	ULTRAMICROTOME RMC PowerTome PC with CR-X Cryosectioning system for preparation of ultrathin section	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	-

Science Case - *TEM investigation of NiMn-based alloys for elastocaloric and magnetocaloric properties*

Here we plan to perform TEM observation on NiMnGa and NiMnTi magnetocaloric and elastocaloric shape memory alloys (SMA) to understand the role of the microstructure on the correlation between martensitic and ferromagnetic phase transformations and mechanical and functional properties.

1. Background and Context

The increasing request of new technological perspectives towards environment sustainability favored in the last years the development of new refrigeration systems to overcome vapor-compression technology, as solid state refrigeration based on multicaloric materials. Shape Memory Alloys are a class of metallic materials which own peculiar functional properties like Shape Memory Effect (SME) and Pseudo-Elasticity (PE) related to solid-solid transition named Thermoelastic Martensitic Transformation (TMT). Different kind of applications that exploit these properties were developed in automotive, aerospace, biomedical and advanced industrial field [1]. Among these smart metals materials, the Magnetic Shape Memory Alloys (MSMA) are widely investigated in the last years thanks to the compresence in these alloys of ferromagnetic transition and TMT. The Heusler NiMn-based ferromagnetic shape memory alloys (FeSMA) have been extensively investigated [2,3] as these materials display a magneto-structural martensitic transformation (TMT) triggered by the application of magnetic field giving an important magnetocaloric effect. In these alloys the Tc and temperatures of TMT are overlapped and the external magnetic field induces a magnetostructural transition involving the high-temperature austenite and the low-temperature martensite, in such a way that the thermal effect is boosted [4]. Besides, the elastocaloric effect (eCE) is a peculiar property of some functional materials in which the heat transfer induced by a simple uniaxial stress can be easily exploited for high-performance cooling applications [5]. The most important materials showing the eCE are the pseudoelastic SMAs and the research activity on this specific topic started about in 2008 [6]. They are promising materials for eCE applications thanks to the thermoelastic martensitic transformation that is induced by an external stress. Indeed, such alloys release heat during loading (exothermic transformation) and absorb heat during unloading (endothermic transformation). The magnetic SMA are good candidates for development of combination of these two caloric effect: magnetocaloric and elastocaloric. Generally, the high brittleness of these systems avoids the achievement of sufficient mechanical properties to open promising elastocaloric functionality. Therefore, in our investigation we concentrate our interest in NiMnGa sintered alloys by Open Die Pressing (ODP) which shown interesting mechanical properties (7). Further a new promising NiMn-based elastocaloric system. NiMnTi. was presented in literature due to theoretical promising High entropy involved in elastocaloric effect [8]. This alloy was prepared in polycrystalline samples and studied. Both systems are interesting reference base for development of new multicaloric materials and the optimization of these alloys requires a deep investigation of microstructure and its correlation with mechanical and functional properties. Particularly a wide diffraction study was performed by temperature-resolved XRD and PDF analysis on NiMnGa ODP samples and attempt to tune the formation of secondary phases in NiMnTi was carried out in correlation with their elastocaloric performances. For these two series of NiMn-based alloys, diffraction structural studies are in progress and a thorough and accurate microscopic observation could be of great help to add important information and to solve the difficult interpretation of diffraction patterns, above all in the martensitic state. Therefore a deep investigation of microstructure and a complete correlation of its cell parameter is worth of study and the TEM investigation is an useful way to have clear demonstration of key microstructural parameters for promising multicaloric SMA alloys. Our research program is based on Self-financed project SMED_ Development of metallic materials and functional devices: Study and characterization of the magnetocaloric and elastocaloric properties of SMA materials for applications in solid-state refrigeration.

Moreover, a PhD study is involved in this investigation.

2. Proposed experiment

- The aim of the experiments is the TEM observation of microstructure of sintered NiMnGa and thermo/mechanically tuned NiMnTi samples. It is interesting to observe the modulation of martensitic state and the parent phase, to individuate possible centre of residual strain, stress concentration and mechanical pinning (e.g. dislocations distribution and stacking faults) for optimized pseudoelastic and magnetic properties.
- TEM is crucial to go inside the punctual microstructure and to highlight the defects, the structural possible distortion and the effect in modulation of martensite and austenite structure, to support the first indication that we are investigating in diffraction pattern registered.
- The registered images will be accurately analysed to individuate microstrutural aspect which are in agreement with the structure indication given from diffraction pattern, or to give further information to guide this study.

3. Summary of previous experimental proposals or characterisation

- No previous experimental proposals or characterizations were performed at ISIS@MACH ITALIA on these samples.
- For these samples we carried out some characterizations which demonstrate the worth of investigation of the particular properties of these alloys, like the stable mechanical behavior for NiMnGa sintered samples [8 and work in progress] and the promising ΔT values measured in elastocaloric cycling of NiMnTi [work in progress]

4. Justification of experimental proposals request

- The TEM is the only microscopic technique able to see in deep the particular microstructure which we would solved and understand
- We ask 5 days to have the sufficient time to try to see two kind of samples (NiMnGa and NiMnTi) in different specimens usefully prepared. In these days is included also the time to properly reduce the thickness of the specimens.

References:

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