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Experiment Proposal

Experiment number GP2024005

Principal investigator (*)	Dr Elena Villa, CNR ICMATE Unit of Lecco, ITALY
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Experiment title	TEM investigation of NiMnTi melt spun ribbons for elastocaloric applications
MRF Instrument	**TEM High Resolution**
Access Route	Direct Access
Science Areas	Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 4
Previous GP Number: GP2023017
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

Shape Memory Alloys, with elastocaloric properties attract interest as caloric materials. Our aim is to investigate their functional properties to apply them in solid state refrigeration. This involves the tuning of the microstructure, by thermomechanical approach and by preparation of specimens with different routes. We obtained interesting caloric properties for polycrystalline NiMnTi alloys, but the need to have specimens more suitable for elastocaloric applications drive us to look for new preparation process as melt spinning. Two series of ribbons are prepared starting from NiMnT18 (at%) and NiMnTi15 (at%) alloys. For these two series the recent functional test on ribbons show interesting elastocaloric properties. But the inverse behavior due to microstructural aspects is not still completely solved and studied. We propose an exhaustive registration of TEM images to catch the microstructural key features which are the origin of this complex mechanical performances.

Publications

Effect of the thermal processing on the microstructural, functional and mechanical properties of cast polycrystalline NiMnTi alloys

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
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Sample record sheet

Principal contact Dr Elena Villa, CNR ICMATE Unit of Lecco, ITALY
MRF Instrument **TEM High Resolution** **Days Requested:** 4
Special requirements:

SAMPLE

Material	NiMnTi alloys ribbon	NiMnTi alloys ribbon	-
Formula	Ni50Mn35Ti15 (at%)	Ni50Mn32Ti18 (at%)	-
Forms	Solid	Solid	
Volume	0.0009 cc	0.0009 cc	
Weight	10 mg	10 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 350 K	300 - 350 K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	no	no	-
Special equip. reqs	none	none	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	none	no	-
Radioactive Hazards	none	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	-

***TEM investigation of NiMnTi melt spun ribbons for elastocaloric applications*:**

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]. The Shape Memory Alloys (MSMA) are widely investigated in the last years like promising materials to the elastocaloric effect (eCE). This 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 [2]. The most important materials showing the eCE are the pseudoelastic SMAs and the research activity on this specific topic started about in 2008 [3]. They are promising materials for eCE applications thanks to the thermoelastic martensitic transformation that is induced by an external stress. Among elastocaloric SMA, NiMnTi was attracted scientific interest due to theoretical promising High entropy involved in elastocaloric effect [4]. In fact, this emerging alloy for elastocaloric purposes is the Heusler all-d-metal NiMnTi which is composed only of elements from the 3d group [5]. This alloy is not characterized by the typical brittleness of the intermetallics, due to the d-d hybridization takes place and the valence electrons are uniformly distributed and generate a metallic bonding [6]. These features give rise to excellent mechanical properties including good ductility and elongation at break [6,7]. Moreover, the large unit cell volume change (ΔV/V0) between the austenitic and martensitic phases that characterizes this alloy induces a high entropy change and therefore good elastocaloric properties [8]. We first worked in development of polycrystalline samples, with thermo-mechanical optimization of microstructure and the polycrystalline state shows peculiar aspect like high hardness and thermal secondary phases segregation. Then polycrystalline ribbons samples were prepared by melt spinning procedure. The samples obtained were first characterized in their functional and mechanical properties and we observed functional aspect as inverse elastocaloric behavior, probably related to internal stress induced by rapid solidification process. This behavior was observed in different kind of melt spun ribbons, and a complete observation and structural demonstration of residual stress/strain pinned in structure has never still reached. Then for these samples it is of great scientific interest diffraction structural studies and a thorough and accurate microscopic observation to add important information and to well individuate the origin and features of the microstructural aspect related to inverse behavior. Moreover, a deep investigation of microstructure and a complete correlation of its cell parameter is worth of study and the TEM investigation is a 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. This proposal gives an important completion of the microstructural investigation which could be fundamental to understand the crucial parameter of microstructure useful for general development for multicaloric purposes. Moreover, a PhD study is involved in this investigation and the series of these samples were prepared during a CNR supported Short Term Mobility in BC Materials at Bilbao in the 2023.

2. Proposed experiment

- *The aim of the experiments is the TEM observation of microstructure thermo-mechanically tuned NiMnTi melt spun ribbons samples, with two Ti content 15at% and 18at%. It is interesting to observe these two compositions to have the possibility to see at RT the martensitic phase stable for Ti15at% and the austenite phase for Ti18at%. Our interest is devoted to individuate possible centre of residual strain, stress concentration and mechanical pinning (e.g. dislocations distribution and stacking faults) for optimized pseudoelastic 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 microstructural 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

- *One previous experimental proposals and characterizations were performed at ISIS@MACH ITALIA on the cast samples of NiMnTi15at% to obtain the complete understanding of crystal structure and modulation of martensite [work in progress]*
- *For these samples we carried out some characterizations which demonstrate the worth of investigation of these particular properties of these systems, like the promising mechanical behaviour and the promising ΔS values measured in elastocaloric cycling [work in progress]*

4. Justification of experimental time requested

- *The TEM is the only microscopic technique able to see in deep the particular microstructure which we would solved and understand*
- *We ask 4 days to have the sufficient time to try to see two kind of samples (NiMnTi15at% and NiMnTi18at%) in different ribbons specimens usefully prepared.*

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