Publication 2

Ge Y., Heikinheimo E., Söderberg O. Lindroos V. K. Microanalysis of a NiMnGa alloy. Proceedings of Scandem2002, (2002), 120-121.

MICROANALYSIS OF A NiMnGa ALLOY

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INTRODUCTION

NiMnGa belongs to a new type of shape memory alloy group. Some of these alloys possess a giant magnetic induced strain, i.e. the MSM-phenomenon, which is strongly dependent on the alloy composition and its crystal structure. In the present work it was studied how a reliable microchemical analysis for NiMnGa can be obtained by SEM-EDS and SEM-WDS. Furthermore, the homogeneity of the samples is discussed on statistical basis.

MATERIALS AND METHODS

A NiMnGa ingot (∅ 20 mm and 100 mm length) was manufactured at Outokumpu Research Oy using modified Bridgman method to obtain as large crystal size as possible. This ingot was homogenised at 1273 K for 72 h and at 1073 K for 48 h in a quartz vacuum ampoule. Two randomly selected samples were made with spark cutting equipment from a horizontal slice of the ingot. They were ground with wet sand papers and electro-polished in 25 % HNO_3-ethanol electrolyte at ambient temperature. For microanalysis, a W-cathode LEO-1450 SEM equipped with Link EDS and WDS was used. Pure Ni, Mn metals and compound GaAs were used as references for elements Ni, Mn and Ga, respectively. The standards were supplied by Micro-Analysis Consultants Ltd.

RESULTS AND DISCUSSION

Microanalysis of the alloy was carried out by using different accelerating voltages from 15 kV to 30 kV with EDS and WDS. Furthermore, the analyses were carried out by using both Ga K_α and Ga L_α lines. Fig. 1 shows the results made from random spots within area of 35×25 μm for Sample 2. According to the degree of scattering, Ga K_α gives consistent results at 20–30 kV with both EDS and WDS. The large scattering of the results at 15 kV is probably due to the poor excitation of Ga K_α-line (9.251 keV, overvoltage ratio ≈1.6). As a result of the absorption correction difficulty for the Ga L-line, Ga K_α gives more reliable result (1). In EDS analysis with Ga L-line, the Ga amount can be overestimated because of the proximity of the Ni L-peak, see Fig. 1. Also, it should be noted the reasonably small difference in the EDS and WDS results with Ga K_α. Consequently, in future one should be able to trust analysis carried out with EDS. The sample homogeneity was investigated by using WDS data, 20 kV, Ga K_α-line. By using limits of $\overline{N} \pm 3\sqrt{\overline{N}}$ for estimating homogeneity, the level of homogeneity was calculated by $(\pm 3\sqrt{\overline{N}} / \overline{N})100$ (%) (2). The results listed in Table 1 show that the samples can be considered homogeneous on micro-scale. According to Table 2, and the results for Mn and Ga, the chemical compositions of Samples 1 and 2 are different.

CONCLUSIONS

Analysing the NiMnGa alloy with K_α line for Ga reliable results at 20-30 kV were obtained with both WDS and EDS. The alloy can be considered homogenous on micro-scale but the two samples do not represent the same composition, i.e. there is macro-scale inhomogeneity in the prepared alloy.

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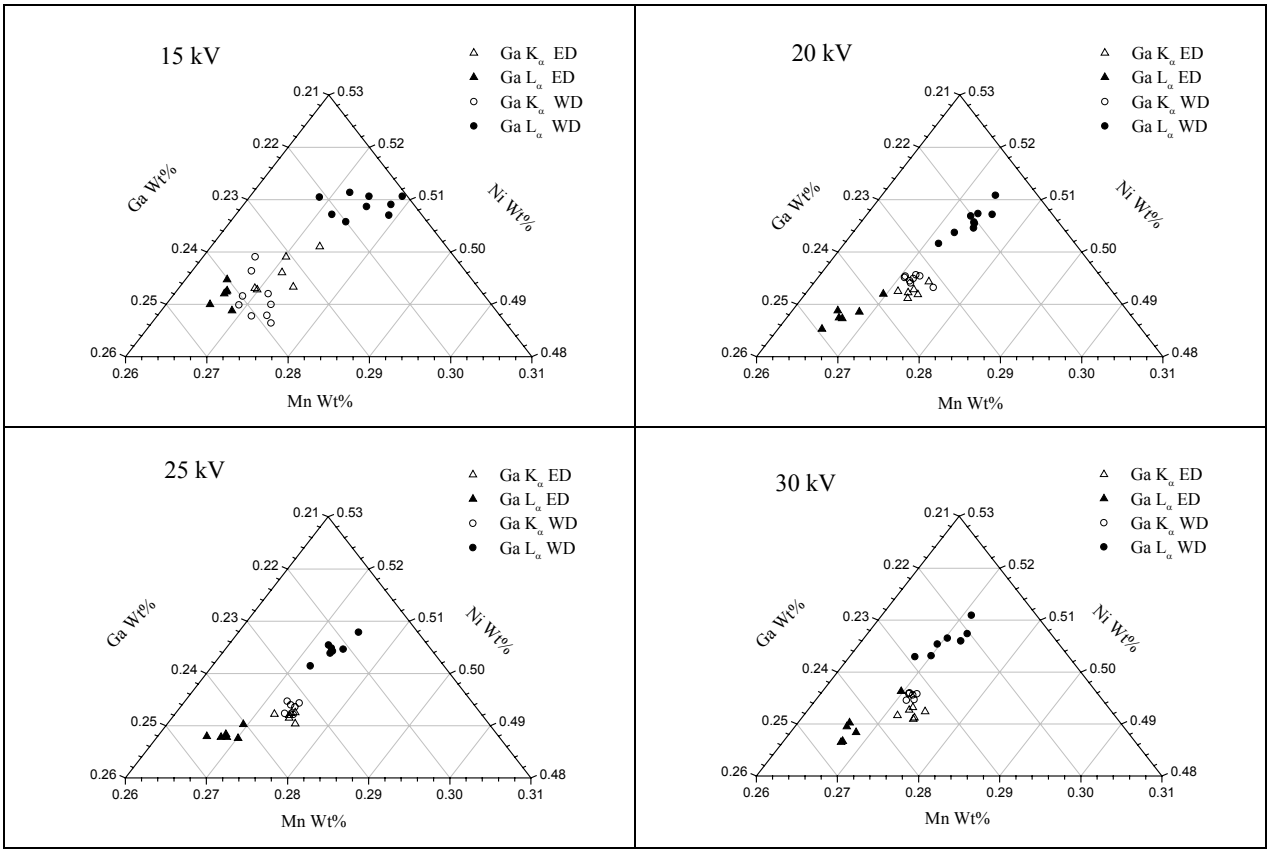

Fig. 1. Ternary composition diagram of Sample 2 measured with EDS and WDS at different beam voltages, using both K- and L-lines for Ga.

Table 1. Calculation of the level of homogeneity, WDS at 20 kV, K_α line for Ga. Beam current for Sample 1 is 46.1 nA, for Sample 2 38.8 nA. Acquisition time for Mn is 15 s, Ni 15 s, and Ga 20 s.

X-ray counts	Sample 1 (10 points)			Sample 2 (8 points)		
	Mn	Ni	Ga	Mn	Ni	Ga
Mean	68779	119832	52858	82039	140439	61145
Max.	69199	120631	53281	82318	141256	61750
Min.	68343	119186	52515	81684	139427	60471
3*sqrt(Mean)	787	1039	690	860	1124	742
High limit	69566	120870	53547	82898	141563	61886
Low limit	67992	118793	52168	81180	139314	60403
Level of homogeneity %	1.14	0.87	1.30	1.05	0.80	1.21

Table 2. The mean values with standard deviations of element analysis for Samples 1 and 2, WDS at 20 kV, K_α line for Ga.

Weight %		Mn	Ni	Ga
Mean	Sample 1 (10 pts)	26.85±0.071	49.46±0.090	23.69±0.092
	Sample 2 (8 pts)	27.20±0.140	49.48±0.087	23.32±0.099