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USING OF ULTRASONIC METHODS FOR DETERMINATION OF THE ELASTIC MODULI ON THE Ti-Ni BASED ALLOYS

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Preliminary Note – Prethodno priopćenje

The presented work is focused on the determination of elastic constants of selected alloys of the systems Ni-Ti and Ni-Ti-Cu with use of ultrasonic methods. Sample preparation was carried out using VIM in graphite crucible in combination with pouring into graphite mould. The prepared samples were appropriately heattreated, polished into the desired shape and subjected to measurement of density and then to ultrasonic measurements. In addition to conventional methods, such as mechanical tests, also less known methods are applied. Among the specific methods we can mention dynamic mechanical analysis, dynamic super microhardness measurement, electro acoustic resonance or ultrasonic test.The group of thus tested materials includes also Ti alloys.

Keywords: Ti-Ni alloys, elastic moduli, ultrasonic methods, alloys, inhomogeneities

INTRODUCTION

Alloys based on NiTi are characterised by shape memory phenomenon generated in a certain temperature interval by reversible martensitic transformation. For practical use of the shape memory effect namely the temperature height and temperature range, in which the martensitic transformation occurs, are very important. They can be affected by the chemical composition and by the choice of technology for the preparation of alloys. Numerous works [1, 2] were already devoted to these issues. Similarly as the determination of physical and mechanical properties it is necessary to know even before the measurement of elasticity moduli the basic structural characteristics of the investigated material, e.g. whether it is a single crystal or polycrystalline material, whether it is homogeneous or heterogeneous in terms of microstructure, its size and shape (e.g. dendrites, grains), present phases, their distribution, etc. Prior to measurement of elasticity moduli it is necessary to know in single crystals the type of lattice and, if possible, also crystallographic orientation in respect to the axis of the sample (usually cylinders). The elasticity moduli can then be determined in the main crystallographic directions, e.g. in the directions [100], [110] and [111]. On the other hand, the physical and mechanical properties at the macro level in the chemically and structurally homogeneous polycrystalline materials are in all directions identical, so it is sufficient to measure and determine the properties in one direction only, i.e. also the elasticity moduli by ultrasound.

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EXPERIMENTAL

Binary and ternary alloys were selected for the experiment. Chemical composition of the alloys is shown in Table 1.

Table 1 **Chemical composition of selected experimental alloys /at %**

Alloy	Chemical composition		
	Ni	Ti	Cu
A	49	50	1
B	51	49	0

The alloys were prepared in a high-frequency vacuum induction furnace in a graphite crucible, fully in accordance with the previously published works [3 - 5]. High purity input materials were used for preparation of the alloys: Ni (C 0,01 %, Fe 0,0048 %, Al < 0,0002 %, Ti 0,0056 %, O_2 0,002 %, N_2 0,0002 %), Ti (C 0,025 %, Fe 0,016 %, Al < 0,002 %, Ni 0,051 %, O_2 0,061 %, N_2 0,0002 %), Cu (99,99 %). Casting of the alloy was performed into a graphite mould, in this manner the ingots with a diameter of 15 mm and a length of 300 mm were prepared. After metallurgical preparation of the alloys swaging at the temperature of 850 °C to the final with diameter of 5 mm was carried out. During forging an inter-stage annealing was performed. The alloys were further examined by optical microscopy (Olympus GX-51 with DP12 camera) and scanning electron microscopy (JEOL JSM 4690 LV). We also performed determination of gases by thermoevolution method (LECO TC 436) and carbon (SpecroMaxx). Using these methods, it was determined that the microstructure contained small carbidic phases of the type TiC of the size of several micrometers. They resulted from the preparation of the alloys in a graphite crucible. The microstructure of

the alloy also contained smaller oxide inclusions of the type oxide inclusions with the size ranging from 0,5 to 1 micrometer. No phases rich in Cu were found in samples of ternary alloys. Samples of cylindrical shape with a height of 6 mm were prepared for next testing. The samples were then annealed at 850 °C for 30 minutes and cooled down in the furnace with an atmosphere of Ar with purity 99,996 %. Bases of cylindrical samples were ground in parallel to the plane. Ultrasonic measurement was performed only in the axial direction. For the measurement of elasticity moduli with use of ultrasound (US) we first determined the samples density ρ / kg.m^{-3}, since the modulus of elasticity is a function of the product of the material density and the square of the US velocity. The density of the samples was determined by hydrostatic method and then longitudinal ($\mathbf{v_L}$) and transverse ($\mathbf{v_t}$) US velocities were measured. These values were measured in the direction perpendicular to the surface of the cylinder bases and to the vector of rotation polarisation of transverse wave in these areas by 360° in order to determine whether there an anisotropy of the US velocity exists in the sample, i.e. whether structural texture also exists in accordance with US measurement [6, 7]. All the measurements were performed in the austenite phase (B2) at room temperature. The alloys with the shape memory phenomenon might have been investigated also within the temperature range of direct and reverse martensitic transformation [8], but this was not realised in the presented work. Under the given temperature conditions and geometrical parameters of the samples these methods work reliably. In order to avoid measurement errors due to spatial inhomogeneities of properties we therefore realised the measurements always on three samples, and the data in tables represent average values. Difference between individual measured values was only minimal.

RESULTS AND DISCUSSION

Metallographic analysis and determination of contents of interstitial elements

Melting was performed with the following parameters. First, the furnace was evacuated to a residual pressure of p = 50 Pa and then filled with Ar (6N) to the pressure p = 20 kPa. This process was repeated 3 times. The furnace was then evacuated to p = 2,5 Pa. Melting was carried out for 10 minutes at the output P = 10 to 19 kW, current I = 100 - 125 A, voltage of 150 - 190 V and a frequency of 3,6 to 3,7 kHz. After melting, the melt was for another approx. 10 seconds stirred by inductive currents. This was followed by filling the furnace with Ar (6N) to the pressure of several kPa and by casting of material. In all castings the content of gas (O_2 and N_2) and of carbon was determined in various parts of the casting, the data in the Table 2 represent average values.

Figure 1 shows the microstructure of ternary alloy A. Microstructure of the alloy A is composed mainly by

Table 2 **Contents of gases in experimental alloys /wt. %**

Alloy	O_2	N_2	C
A	0,0754	0,052	0,034
B	0,0638	0,046	0,025

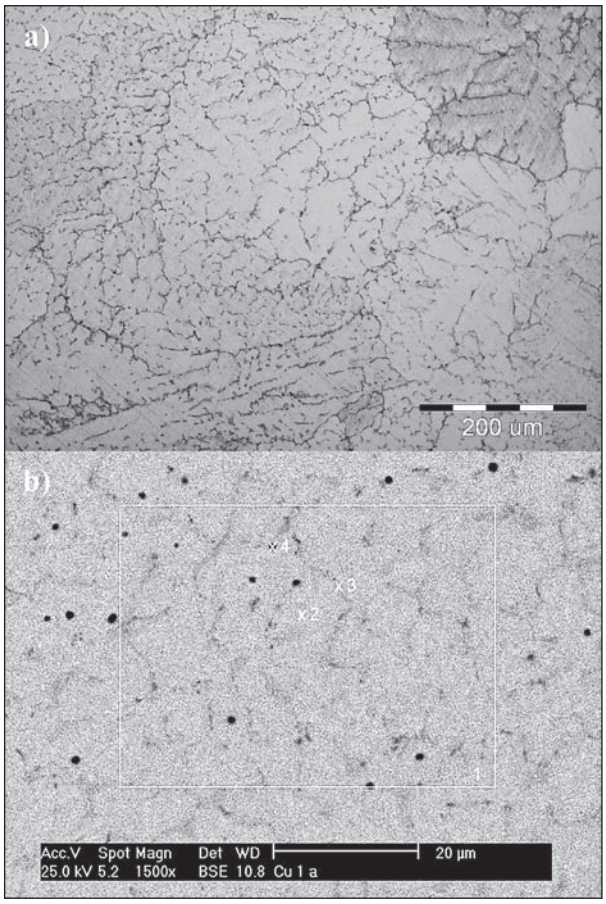

Figure 1 Microstructure of alloy A, as cast, a) optical micrograph, cross section 200x; b) SEM-BSE image

a TiNi intermetallic compound (Region 2 in Figure 1b), in which a larger amount of Cu is dissolved. We can see in the microstructure also darker smaller longitudinal formations corresponding to the phase Ti_2Ni. They contain a smaller amount of Cu compared with the TiNi phase. There are also minor carbidic TiC type formations, which correspond in Figure 1 to the Area 4. The area 1 shows the area for indicative determination of the chemical composition of the alloy by the EDX method. Chemical composition of the alloy was confirmed by this method.

Figure 2a shows the microstructure of the binary alloy B in the as cast condition, it is a cross-sectional view. The microstructure of the alloy in the as cast state is very similar to the microstructure of the alloy A. As it can be seen in Figure 2b, it is again formed by the majority TiNi intermetallic phase (area 1), in which the darker areas are formed by the Ti_2Ni phase. We can see here also a significant amount of carbidic phases of the TiC type of similar size as that of the alloy A (area 4). The area 1 (marked by rectangle) indicates the area used for indicative determination of the alloy chemical composition. In this case too, the chemical composition was confirmed by the EDX method.

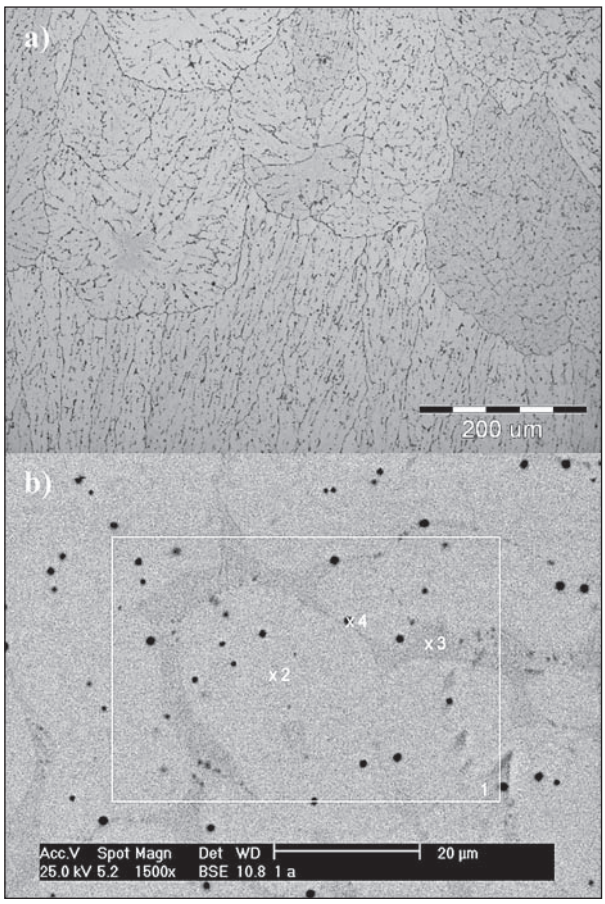

Figure 2 Microstructure of alloy B, as cast, a) optical micrograph, cross section 200x; b) SEM-BSE image

Ultrasonic analysis

The US method was applied on several materials among others Ti based [9 - 11]. In spite of this fact the results that could be used for direct comparison with this study are only rare. It is generally known that tensile tests seem to give slightly lower Young's modulus (B) values than US methods. Table 3 summarises the results of the measurements of specific characteristics for the alloys A and B. The modules of elasticity (E) were calculated from the average rates of US, presented in table 3 according to the following equations:

$$E = \rho v_L^2 (3a^2 - 4)/(a^2 - 1), \quad a = v_L/v_t \qquad (1)$$

$$G = \rho v_t^2 \qquad (2)$$

$$B = \rho(3v_L^2 - 4v_t^2) \qquad (3)$$

$$= (a^2 - 2) / 2(a^2 - 1) \qquad (4)$$

It is evident from the presented table that although the density of individual types of alloys differs only negligibly, situation is different what concerns the modulus of elasticity (E) and Poisson's ratio (). The difference of 4 GPa between the samples A and B (Table 3) can serve as concrete example of this. The difference in the values of elastic modules and in particular the difference in the values of the Poisson's ratio of transverse contraction can already be considered significant and it might have been invoked either by different constitution, or by the change of the physical-metallurgical parameters during preparation of the samples. It is highly probable that comparatively high value of may be reflected also in the parameters of the shape memory effect of NiTi alloys.

Table 3 **The measured values of density ρ, v_L longitudinal and transverse US velocity v_t, modulus of elasticity E, shear modulus G, Young's modulus in a versatile pressure B, and Poisson's ratio**

Alloy	ρ / kg·m^{-3}	v_L / m·s^{-1}	v_t / m·s^{-1}
A	6407,4	5201,9	1878,0
B	6467,8	5301,5	1803,5
E / GPa	G / GPa	B / GPa	
64,42	22,61	143,24	0,425
60,37	21,05	153,72	0,435

CONCLUSIONS

Experimental material with quite satisfactory characteristics was prepared by vacuum induction melting. Satisfactory levels of interstitial impurities were found, as presented in Table 2. Using the techniques of optical and scanning electron microscopy we performed microstructural analysis, which established that both alloys were homogeneous, free from casting defects, and the required chemical composition was confirmed by the EDX technique. The microstructure contained only small areas of the phase Ti_2Ni and also carbidic phases of the type TiC (which resulted from the preparation of experimental material in a graphite crucible). The samples for the next experiment were successfully prepared by swaging. The results of determination of the density of both alloys (ternary and binary) did not show any substantial differences, the determined density of the alloys therefore corresponded to the current state of knowledge. The measured values of elastic constants in the austenitic state slightly differed for both alloys (as shown in Table 3), it can be said, however, that the determined values correspond to the current state of knowledge in this field.

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Note: The responsible translator for English language is Boris Škandera, Ostrava, Czech Republic