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**MICROSTRUCTURAL CHARACTERIZATION OF THE NEW TOOL Ni-BASED ALLOY WITH HIGH CARBON AND CHROMIUM CONTENT****CHARAKTERYSTYKA MIKROSTRUKTURY STOPU MODELOWEGO MATERIAŁU NARZĘDZIOWEGO NA OSNOWIE Ni O DUŻEJ ZAWARTOŚCI WĘGLA I CHROMU**

The concept of new tool materials, based on Ni alloys strengthened by intermetallic compounds, intended for applications at high temperatures is presented in the paper. The chemical composition of this new material was designed in such a way as to obtain the matrix strengthening by means of the precipitation of intermetallic compounds rich in Ni and to have the carbide fraction above 25%. Carbides should remain stable in the microstructure, regardless of the heat treatment, since they favourably influence an abrasion resistance. The results of microstructure investigations in the as-cast condition, are given. The type of phases appearing in the microstructure was determined and their morphology described. The main microstructure components of the investigated Ni-based alloy with high carbon and chromium content are: the  $\gamma$  phase, which constitutes a matrix, the  $\gamma'$  phase, which occurs as fine globular precipitates and the primary Ta and Cr carbides (of MC and  $M_7C_3$  type – respectively).

*Keywords:* tool materials, Ni based alloys, intermetallic compounds, carbides

W pracy przedstawiono koncepcję nowych materiałów narzędziowych na osnowie niklu umacnianych związkami faz międzymetalicznych, przewidywanych do pracy w wysokich temperaturach. Skład chemiczny nowego materiału zaprojektowano tak, aby umocnienie osnowy uzyskać w wyniku wydzielenia związków metalicznych bogatych Ni, a udział węglików wynosił ponad 25%. Węgliki powinny pozostać stabilne w mikrostrukturze niezależnie od obróbki cieplnej, wpływając korzystnie na odporność na ścieranie. Zamieszczono badania mikrostruktury stopu w stanie lanym. Określono rodzaj faz występujących w mikrostrukturze oraz opisano ich morfologię. Głównymi składnikami mikrostruktury badanego stopu na osnowie Ni, o dużej zawartości węgla i chromu są: faza  $\gamma$ , która stanowi osnowę, faza  $\gamma'$ , która występuje w postaci drobnych kulistych wydzieleni oraz pierwotne węgliki Ta (typu MC) i Cr ( $M_7C_3$ ).

**1. Introduction**

An increased interest of the machine elements production from advanced materials (such as high alloyed steels or Ti alloys) forces the development of tool materials for their treatment, especially for forming at high temperatures.

Tool steels containing from 0.30 to 0.60% C, up to 5% Cr and Mo, W and V are universally applied as tool materials for operations at high temperatures. Tools made of these steels obtain functional qualities by means of a heat improvement, it is by combining quenching procedures with medium or high tempering. Tempering of tool steels is usually done in the temperature range: 550÷ 620°C. Strengthening is achieved by precipitating

alloy carbides of MC and  $M_2C$  (V, Mo and W) type [1÷5].

Several tools have to operate at temperatures above 600°C, sometimes even at 1000°C, at which quenched and tempered steels soften, causing that a lifespan of tools rapidly decreases.

The chemical composition of tool steels was, for many years, modified to improve their hot-working properties. The complex alloys Cr-Ni-Co-Fe with additions of W, Mo, Nb, in which a significant part of iron was substituted by Co, were developed [6]. A group of alloys based on the Co matrix (Stellites) having good tribological properties intended for cutting tools was obtained. These alloys can be divided into certain main groups: Co-Cr-W-C and Co-Cr-W/W-Ni/Fe-C with additions of Si+B [7,8]. Unfortunately the maximum temperature

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range in which those alloys can operate is  $600\div 750^{\circ}\text{C}$  only.

A development of high temperature creep-resisting nickel-based alloys was mainly the modification of 80% Ni and 20% Cr alloy known for its good creep-resistance. On account of ineffectiveness of strengthening by carbides in high temperatures a hardening of Ni-based alloys was obtained by the intermetallic compound  $\text{Ni}_3(\text{Ti}, \text{Al})$  designated as  $\gamma'$  [9,10].

Several alloys were developed on the concept of Ni-based matrix strengthened by  $\gamma'$  phase, among others, the alloys of an increased carbon content and a complex chemical composition [11÷12].

There are known applications of Ni-based superalloys such as IN617, RR1000 [13, 14] or alloys of a complex composition [15] for tools operating at high temperatures. However, a carbon content in such alloys is quite low (not exceeding 0.1%) and obtaining a large fraction of a carbide phase – which would allow to achieve the good tribological properties of tools – is not possible.

The determination of microstructure components of the newly designed tool Ni-based alloy strengthened by intermetallic phases – expected for applications at high temperatures – is the primary purpose of the presented paper.

## 2. Experimental procedure

The chemical composition of the new Ni-based alloy was designed in the Laboratory of Phase Transformations, Department of Physical and Powder Metallurgy, AGH University of Science and Technology.

The microstructure of the investigated material was examined by the light microscope Axiovert 200 MAT and the scanning electron microscope FIB Zeiss NEON 40EsB CrossBeam.

X-Ray phase analysis was performed by means of the Diffractometer D500 of the Siemens Company, using filtered radiation of a copper anode lamp.

The hardness measurements were performed with the Vickers HPO250 apparatus.

The carbon content was measured using the LECO CS-125 analyser.

## 3. Material for investigations

The chemical composition of the investigated alloy (Table 1) was designed in such a way as to obtain the matrix strengthening by precipitations of a metallic phase rich in Ni accompanied by a high carbide fraction. Carbides should remain stable in the microstructure – regardless of the heat treatment – since they favourably

influence the abrasion resistance. It was assumed, when designing the alloy composition, that the primary Ta carbides of MC type will be formed. The Ta content was selected to bind carbon into a carbide form and to form the  $\gamma'$  phase together with Al and Ni. Zirconium was added to harden grain boundaries while chromium to increase the heat resistance. The Ni matrix was chosen due to the lack of allotropic transformations, which could destabilise the microstructure and properties during a hot-working exploitation. Because of patent pending properties of the investigated alloy, contents of Ta, Al and Cr are not given precisely.

TABLE  
Chemical composition (wt.%) of the investigated alloy

C	Ta	Al	Cr	Zr	P	S	Ni
0.84	x	x	<12	0.2	0.01	0.01	Bal.

A test melt of a mass of approximately 1 kg was made in a vacuum furnace, and cast into a ceramic mould. Casting together with the first cut sample (as an example) is shown in Figure 1. Samples were cut from the casting foot. Examinations were made on polished sections parallel and perpendicular to the casting surface.



Fig. 1. View of the investigated alloy casting

#### 4. Results and discussion

Microstructures of the examined alloy in as-cast state obtained by means of the light and scanning microscope are presented in Figs 2 and 3 – respectively. Materials were of large grains, characteristic for as-cast state, inside which dendritic zones were revealed (Figs. 2a-c). Tantalum carbides of MC type and chromium carbides  $Cr_7C_3$  are distributed in interdendritic zones (shown at larger magnifications in Figures 2d, 2e and 3). Tantalum carbides are shown at large magnifications in Figure 3c. Ta and Cr carbides were identified by the EDS analysis (Figs 4a-c) and the X-Ray phase analysis (Figs 5a,b and e). The carbide volume fraction (app. 33%) was estimated by the point-count method. Primary carbides of irregular longitudinal shapes and various sizes are uniformly

distributed, not forming any agglomerates. Nevertheless, contrary to the described in paper [16] alloy with titanium, where carbides occurred in the total volume of the material (compare Fig. 6), they were in interdendritic zones in the investigated alloy. No presence of graphite, as in the model alloys described in papers [17,18], was found (compare Fig. 7). Apart from the carbides, fine precipitates of the intermetallic phase in matrix  $\gamma$  – of such small size that its accurate identification by the EDS analysis was impossible – are shown in photographs from the scanning electron microscope (Fig. 3d). This phase is rich in nickel, aluminium and tantalum. It was confirmed, by the X-Ray phase analysis, that this is the  $\gamma'$  phase (Fig. 5d), the most probably  $Ni_3AlTa$ , although Cr as its component is also possible.

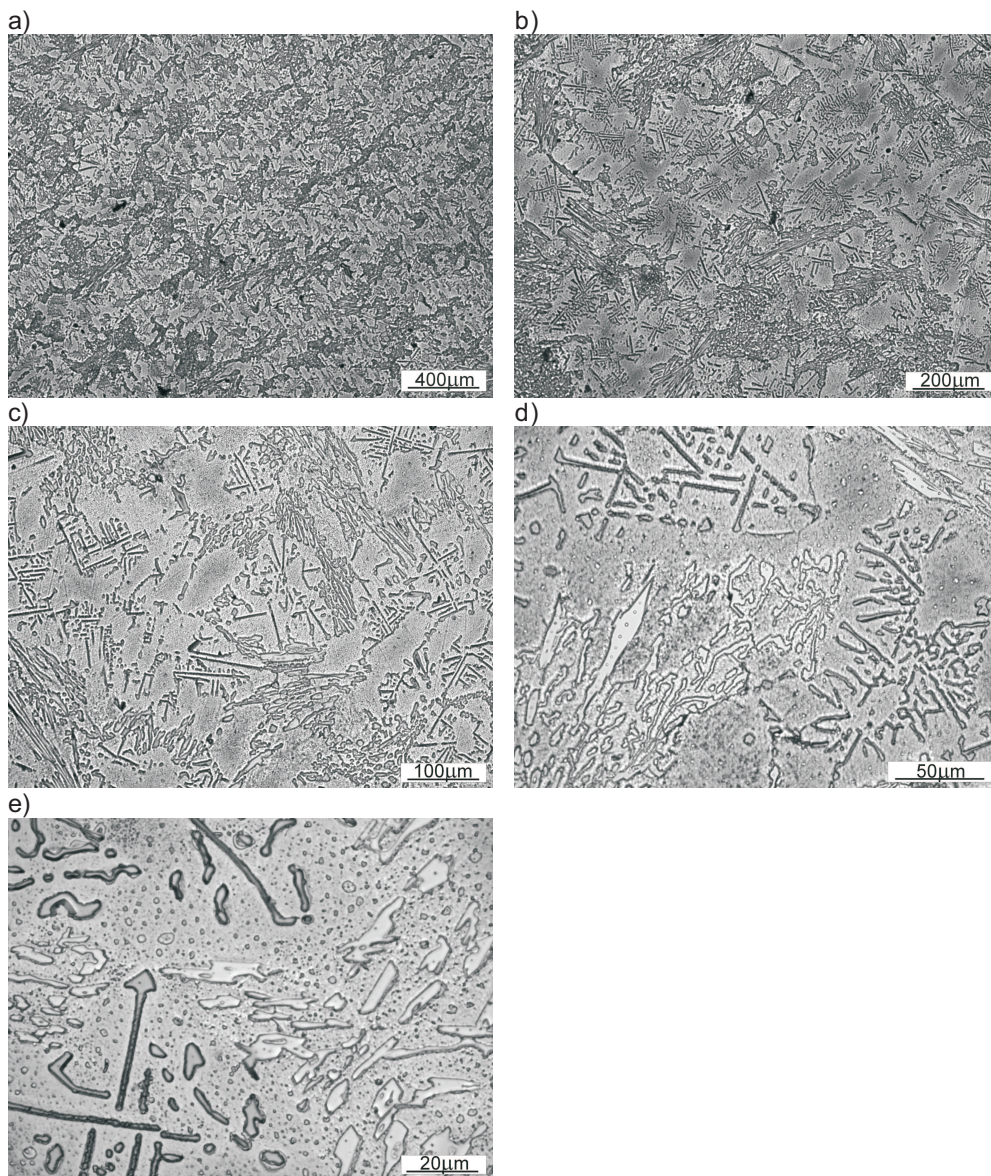


Fig. 2. Microstructure of investigated alloy in as-cast state. Light microscope

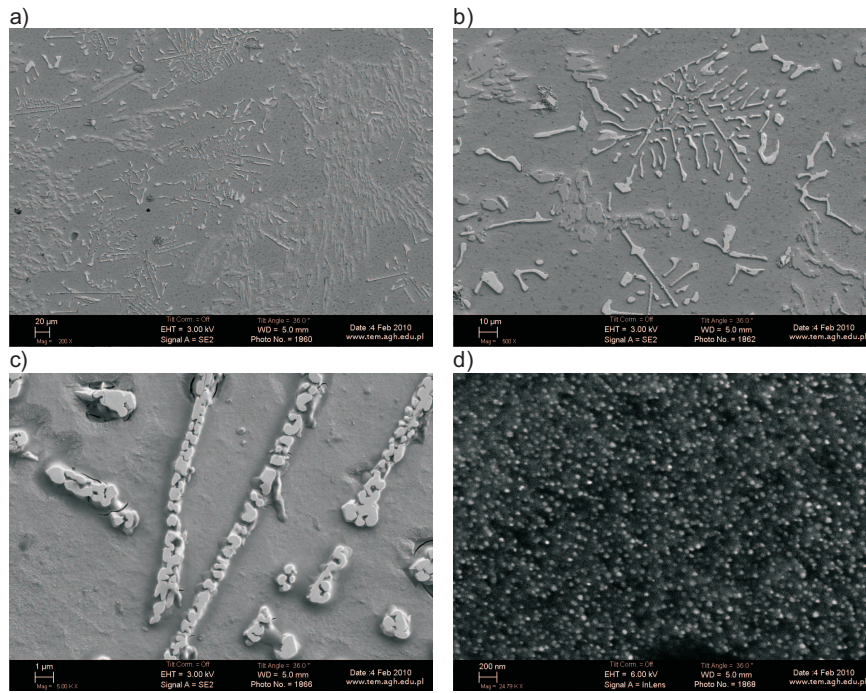


Fig. 3. Microstructure of investigated alloy in as-cast state. SEM

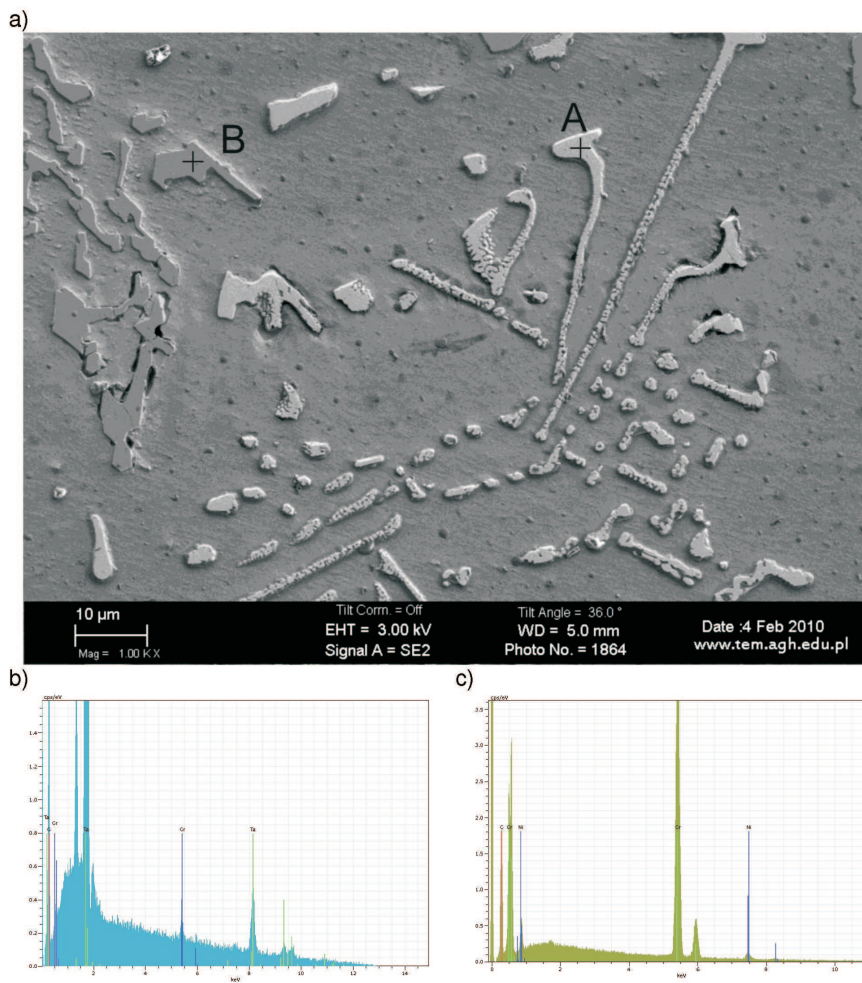


Fig. 4. a) Microstructure of the investigated alloy in as-cast condition with marked zones where from the EDS analysis was performed; b) Characteristic spectrum from A zone; c) Characteristic spectrum from B zone

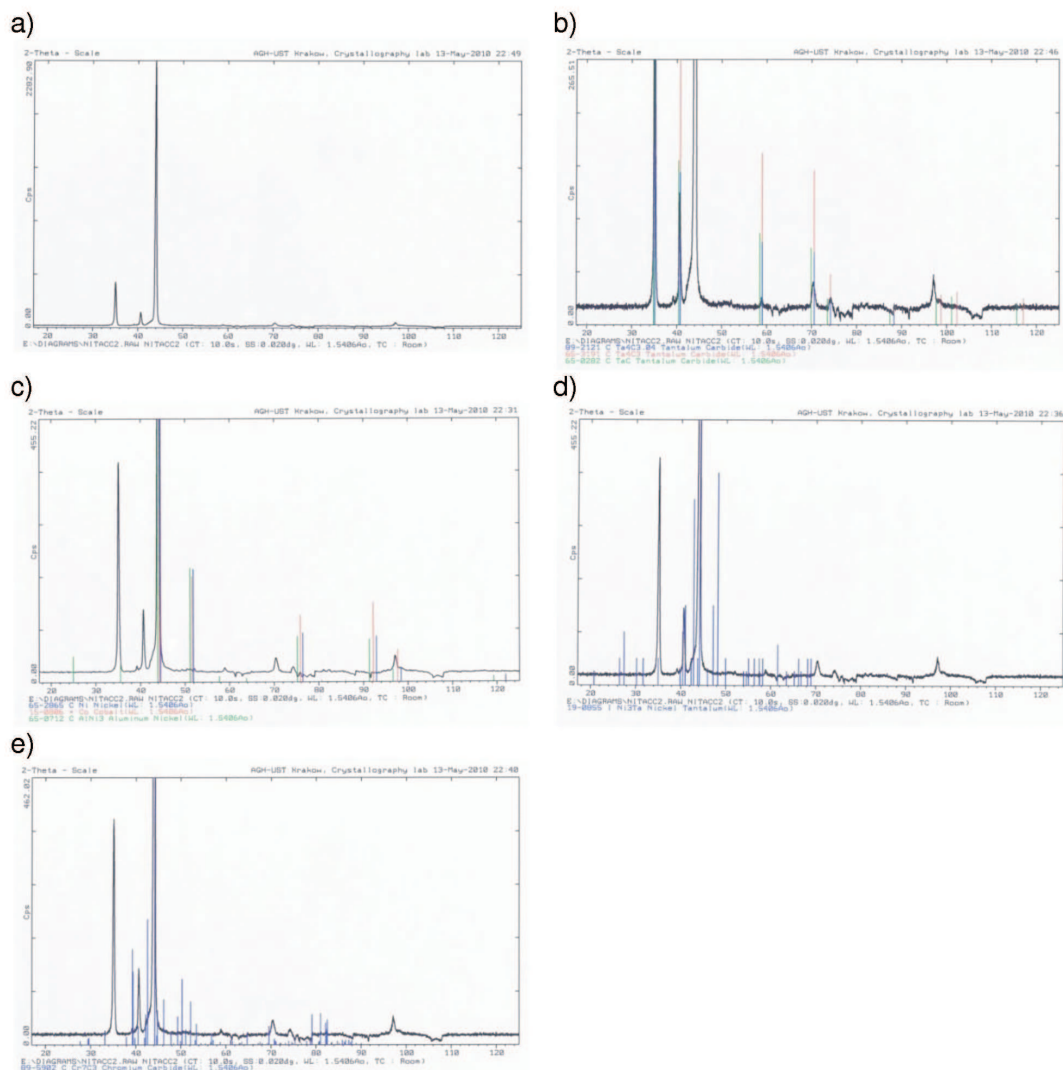


Fig. 5. a) Diffractogram, b) Diffractogram from Figure a – with lines characteristic for TaC and Ta<sub>4</sub>C<sub>3</sub>carbides, c) Diffractogram from Figure a – with lines characteristic for Ni, Co and Ni<sub>3</sub>Al, d) Diffractogram from Figure a – with lines characteristic for Ni<sub>3</sub>Ta, e) Diffractogram from Figure a – with lines characteristic for Cr<sub>7</sub>C<sub>3</sub> carbide

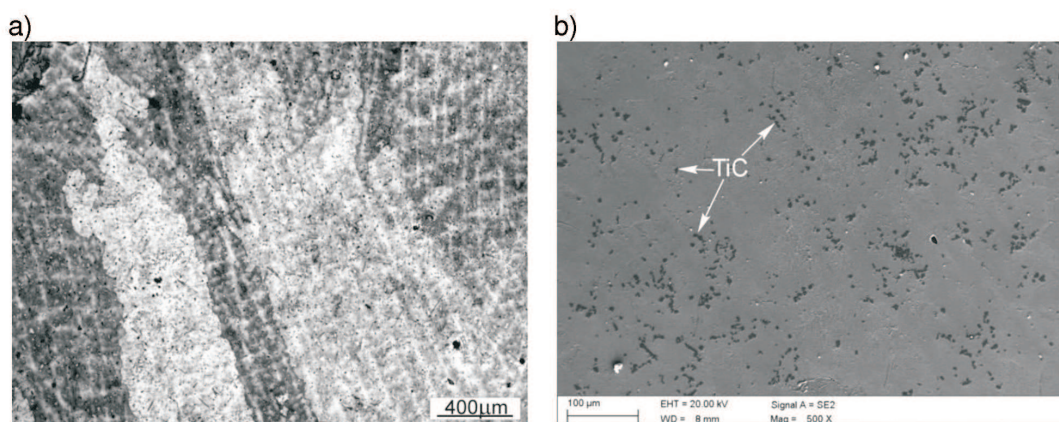


Fig. 6. Microstructure of the Ni-based alloy with Ti and Fe: a) Morphology of grains from the casting in the light microscope; b) Morphology of carbides, SEM [16]

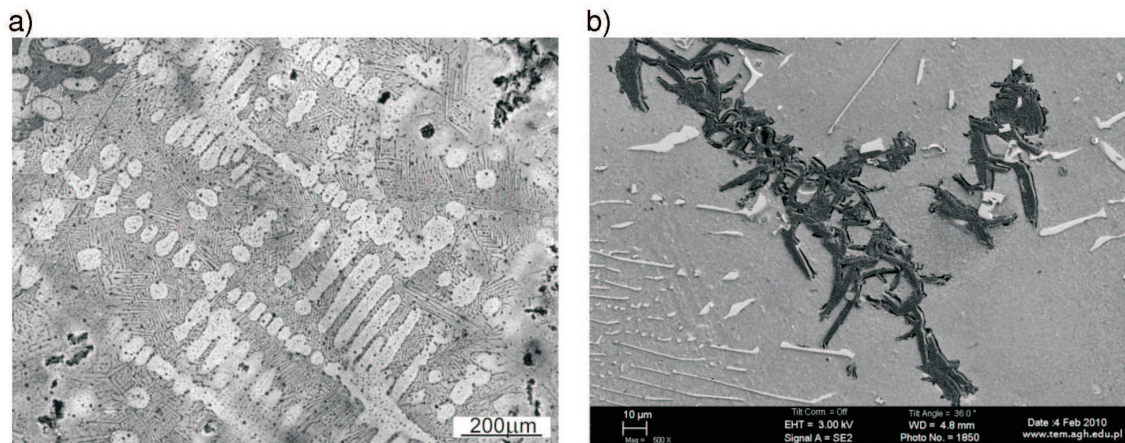


Fig. 7. Microstructure of the Ni-based model alloy with Ta and Co: a) Area of some grains, the light microscope, b) Graphite precipitations, SEM [17]

Hardness measurements were performed for samples taken from various places on an ingot cross-section. Hardness measured at the casting surface equals 355 HV10 and increases in the casting axis direction to 366 HV10. This is the result of the alloy elements segregation before the solidification front, nonetheless hardness differences are not large.

It should be emphasised that the high purity alloy formation was succeeded. This alloy does not contain neither sulphides or zones of the  $\gamma/\gamma'$  eutectic characteristic for Ni-based alloys in as-cast condition. Such eutectic due to its brittleness is an undesired component. The  $\gamma$  phase, which is a solution of alloying elements in Ni matrix (Fig. 5d) constitutes the matrix of the investigated alloy.

Further examinations will be carried out in order to estimate the carbide phase stability, to select the optimal heat treatment and to determine tribological properties at low and high temperatures.

## 5. Conclusions

Microstructure of the investigated alloy in as-cast state consists of:  $\gamma$  phase, which constitutes the matrix,  $\gamma'$  phase, which occurs as fine globular precipitates and primary tantalum (MC type) and chromium ( $\text{Cr}_7\text{C}_3$ ) carbides. Primary carbides of irregular shapes are distributed in interdendritic zones. The assumed volume fraction of the primary carbides was achieved.

As the result of the alloy elements segregation before the solidification front, hardness on the casting cross-section changes, nonetheless the hardness differences are not large. Further heat treatment should eliminate these differences.

The graphite presence was not found, which indicates the proper balance of carbon and carbide forming elements content and the proper selection of solidification conditions. The alloy did not contain sulphides and zones of the  $\gamma/\gamma'$  eutectic – characteristic for Ni-based alloys in as-cast condition.

## Acknowledgements

The authors would like to thank Prof. J. Pacyna and Prof. A. Czyrska-Filemonowicz from AGH University of Science and Technology, Krakow for the valuable help in this research.

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