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MICROSTRUCTURE CHARACTERIZATION OF HIGH CARBON ALLOY FROM THE Ni-Ta-Al-Co-Cr SYSTEM**CHARAKTERYSTYKA MIKROSTRUKTURY WYSOKOWĘGLOWEGO STOPU Z UKŁADU Ni-Ta-Al-Co-Cr**

In the present work results of investigations of the new high carbon alloy from the Ni-Ta-Al-Co-Cr system are presented. The alloy has been designed to have a good tribological properties at elevated temperatures. The chemical composition of this material was designed to obtain a matrix strengthening by the precipitation of γ' phase ($\text{Ni}_3(\text{Al,Ta})$) and the primary carbides volume fraction above 25%. The primary carbides should remain stable in the microstructure, regardless of the heat treatment, in order to increase a wear resistance. The results of microstructure investigations in the as-cast condition are presented. 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, cobalt and chromium content are: the γ phase, which constitutes a matrix, the γ' phase, which occurs as fine globular precipitates and the primary Ta and Cr carbides (of MC and M_7C_3 type – respectively).

Keywords: Ni-based alloys, gamma' phase, carbides, tantalum, chromium

W pracy przedstawiono nowy stop na osnowie niklu o dużym stężeniu węgla, przewidywany do pracy w wysokiej temperaturze i w warunkach silnego zużycia tribologicznego. Skład chemiczny nowego materiału zaprojektowano tak, aby umocnienie osnowy uzyskać w wyniku wydzielania fazy γ' ($\text{Ni}_3(\text{Al,Ta})$), a udział węglików pierwotnych wynosił ponad 25%. Węgliki powinny pozostać stabilne w mikrostrukturze niezależnie od obróbki cieplnej, wpływając korzystnie na odporność na ścieranie. Zamieszczono wyniki badań 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, kobaltu i chromu są: faza γ (która stanowi osnowę) faza γ' (która występuje w postaci drobnych kulistych wydzieleni) oraz pierwotne węgliki Ta (typu MC) i Cr (M_7C_3).

1. Introduction

The microstructure and properties of tools have strong influence on their throughput and reliability what favors development of mechanization and automate of technological lines. An increased interest of the machine elements production made of advanced materials (such as high alloyed steels or titanium alloys) forces the development of tool materials for their forming at high temperatures.

Steels are a typical material for tools production. For high temperatures application a hot-work and high speed steels are applied [1÷5]. The hot work 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. Usually, the most important property of the steel is high impact resistant, which is required to crack resistant. Therefore their microstructures do not

include a primary or secondary carbides. Tools made of these steels obtain functional qualities by toughening, i.e. combined quenching with medium or high tempering. Tempering of tool steels is usually in the temperature range: 550÷620°C. Strengthening is achieved by precipitation of alloy carbides of MC and M_2C (V, Mo and W) type [1,2].

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 lifetime of tools rapidly decreases.

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 $\text{Ni}_3(\text{Ti, Al})$, designated as γ' [6,7].

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Several alloys were developed on the concept of Ni-based matrix strengthened by γ' phase, among others, the alloys of an increased carbon content and a complex chemical composition [8,9].

There are known applications of Ni-based superalloys such as IN617, RR1000 [10, 11] or alloys of a complex composition [12] for tools operating at high temperatures. However, a carbon content in such alloys is quite low (not exceeding 0.1%) and obtaining a large volume fraction of carbides, which would allow to achieve the good tribological properties of tools, is not possible.

The determination of microstructure components of the new designed high carbon Ni-based alloy strengthened by intermetallic γ' phases, expected for applications at high temperatures and at strong wear condition, is the primary purpose of the presented paper.

2. Experimental procedure

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 by use of 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 to obtain the matrix strengthening by precipitations of a γ' phase ($\text{Ni}_3(\text{Al,Ta})$) accompanied by a high primary carbide fraction. Primary carbides should remain stable in the microstructure, regardless of the heat treatment, in order to increase the wear resistance. It was assumed, that the primary tantalum carbides of MC type will be formed. The tantalum content was selected to bind carbon into a carbide form and to form the γ' phase together with aluminium and

nickel. Zirconium was added to harden grain boundaries while chromium and cobalt 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. Because of patent pending properties of the investigated alloy, chemical composition is not given precisely.

TABLE 1
Chemical composition (wt. %) of the investigated alloy

C	Ta	Al	Cr	Co	Zr	P	S	Ni
0.898	x	x	>12	20	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. Samples were cut from the casting foot. Examinations were made on polished sections parallel and perpendicular to the casting surface.

4. Results and discussion

Microstructures of the investigated alloy in as-cast state obtained by the light and scanning microscope are presented in Figs 1 and 2 – respectively. Material was of large grains, characteristic for as-cast conditions, inside which dendritic zones were revealed (Fig. 1a). Tantalum carbides of MC type and chromium carbides Cr_7C_3 are distributed in interdendritic zones (shown at larger magnifications in Fig. 2a). Ta and Cr carbides were identified by the EDS analysis (Figs 3a-c) and the X-Ray phase analysis (Fig. 4). The carbide volume fraction (app. 47%) was estimated by the point-count method. The volume fraction of primary carbides of the investigated alloy from Ni-Ta-Al-Co-Cr system, is higher than of high carbon alloy from the Ni-Ta-Al-Cr system, which was described in paper [13]. The difference is about 12%. The difference in chemical composition of these two alloys is only with cobalt addition. It means that cobalt causes alloy elements segregations during solidification. The carbon, tantalum and chromium concentration in interdendritic zones in Ni-Ta-Al-Co-Cr alloy are high and finally the volume fraction of primary carbides increase.

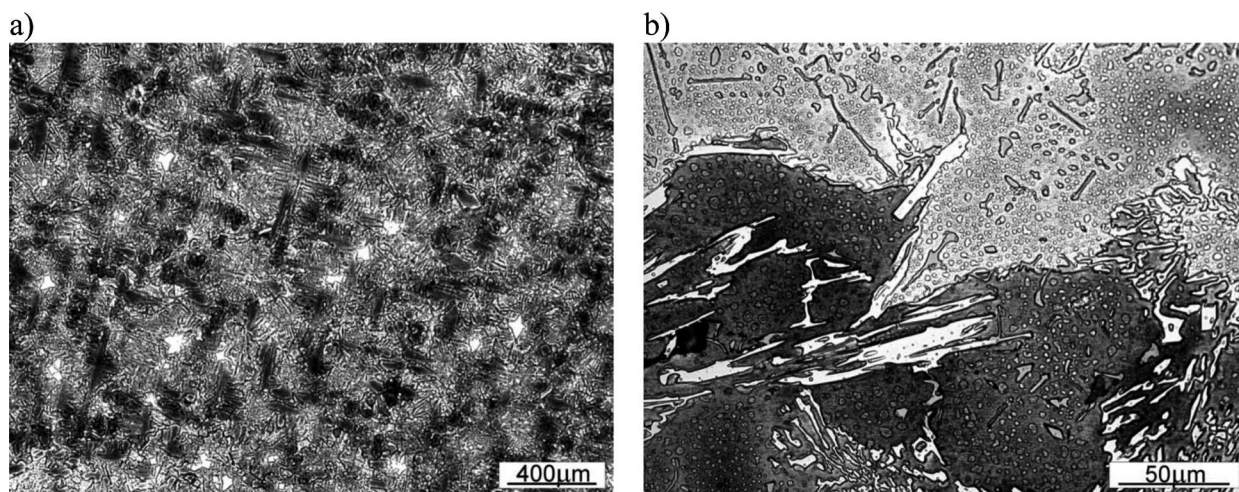


Fig. 1. Microstructure of investigated alloy in as-cast state: a) dendritic microstructure, b) chromium (light) and tantalum primary carbides. Light microscope

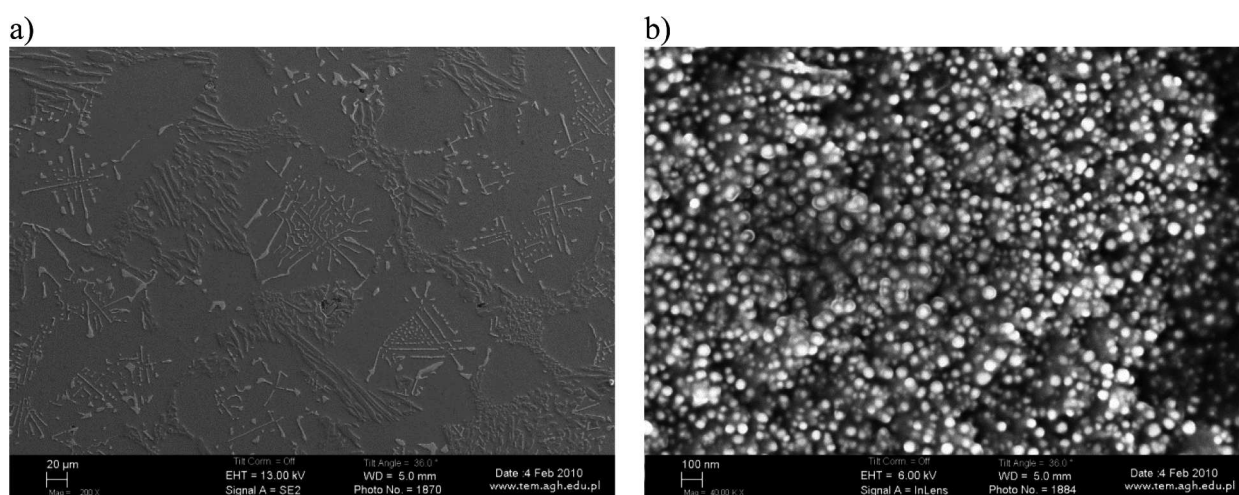


Fig. 2. Microstructure of investigated alloy in as-cast state. a) carbides morphology, b) morphology of γ' phase. SEM

Primary carbides of irregular longitudinal shapes and various sizes are uniformly distributed, do not form any agglomerates. The morphology of tantalum carbides is like Chinese script. Contrary to the described in paper [14] alloy with titanium, where carbides occurred in the total volume of the material, they were in interdendritic zones in the investigated alloy. No presence of graphite, as in the high carbon alloy from Ni-Ta-Al-Co system described in papers [15], was found. Apart from the carbides, fine precipitates of the intermetallic phase in γ matrix – of such small size (about 50 nm) that its accurate identification by the EDS analysis was impossible – are shown in photographs from the scanning electron microscope (Fig. 2b). This phase is rich in nickel, aluminium and tantalum. It was confirmed, by the X-Ray phase analysis, that this is the γ' phase, the most probably $\text{Ni}_3(\text{Al},\text{Ta})$.

Hardness measurements were performed for samples taken from various places on an ingot cross-section. Hardness measured at the casting surface was 350 HV10 and increases in the casting axis direction to 360 HV10. This is the result of the alloy elements segregation before the solidification front and different solidification condition, nonetheless hardness differences are negligible.

The investigated alloy does not contain neither sulphides nor zones of the γ/γ' eutectic characteristic for Ni-based superalloys in as-cast condition. A solid solution of cobalt, chromium, aluminium and tantalum in nickel (Fig. 3d) 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.

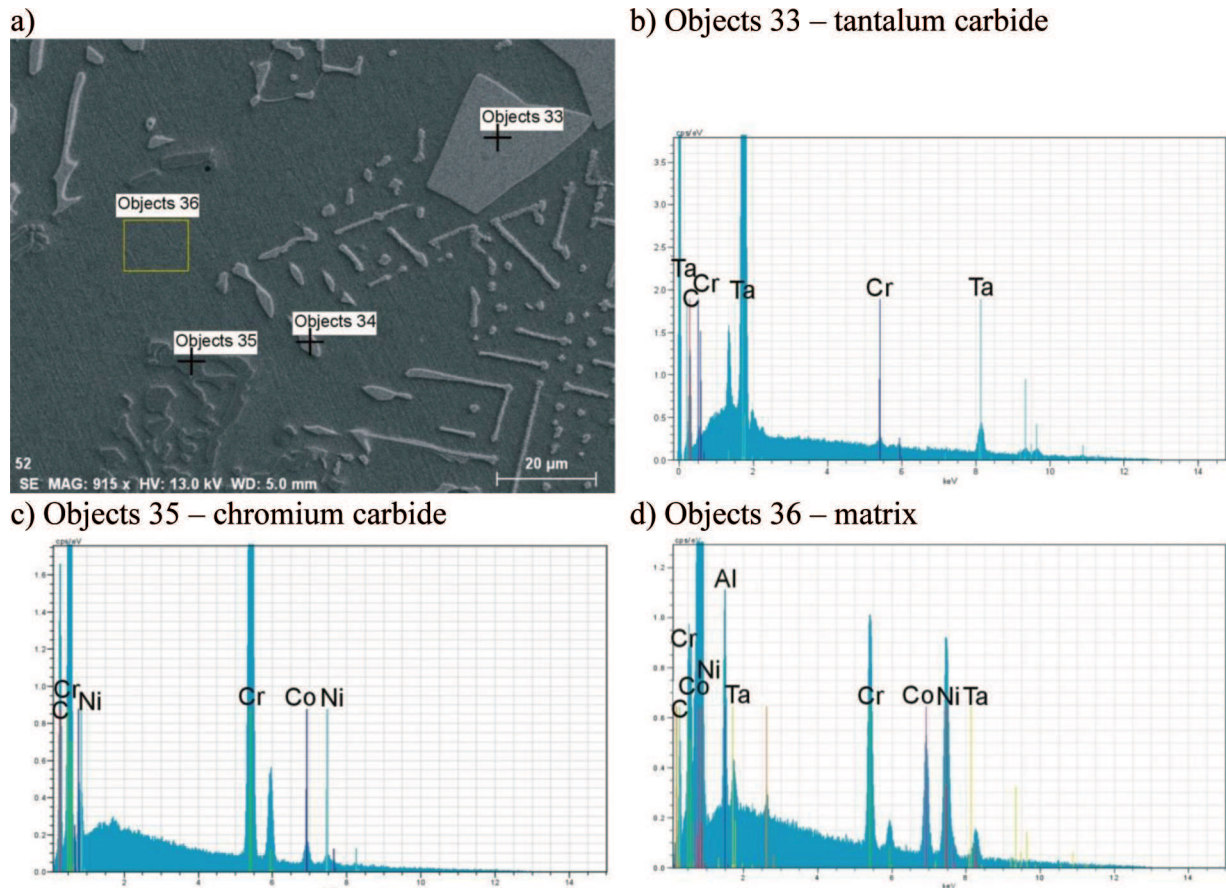


Fig. 3. a) Microstructure of the investigated alloy in as-cast state with marked zones where from the EDS analysis was performed; b) Characteristic spectrum from objects 33; c) Characteristic spectrum from objects 35; d) Characteristic spectrum from objects 36

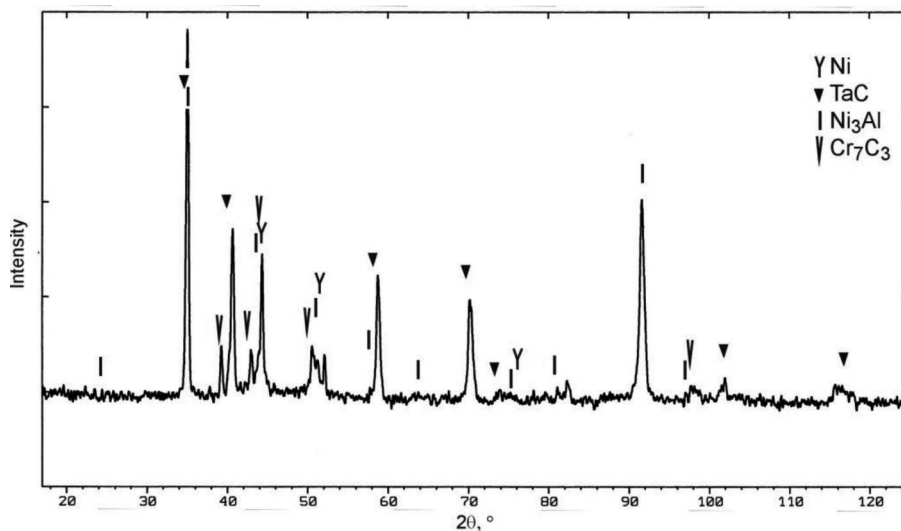


Fig. 4. X-ray diffraction of investigated alloy in as-cast condition

5. Conclusions

1. Microstructure of the investigated alloy in as-cast state consists of: γ phase, which constitutes the matrix, γ' phase, which occurs as fine globular precipi-

tates and primary tantalum (MC type) and chromium (Cr_7C_3) carbides.

2. The volume fraction of primary carbides in the alloy of the Ni-Ta-Al-Co-Cr system, is high (47%). Primary carbides of irregular shapes are distributed

in interdendritic zones. The morphology of tantalum carbides is like Chinese script.

3. Cobalt addition into Ni-Ta-Al-C-Cr alloy causes other alloy elements segregations during solidification. The carbon, tantalum and chromium concentration in interdendritic zones are high and finally the volume fraction of primary carbides is high.
4. The graphite presence was not found, which indicates the proper balance of carbon and carbide forming elements content as well as the proper selection of solidification conditions. The alloy did not contain sulphides and zones of the γ/γ' eutectic – characteristic for Ni-based alloys in as-cast condition.

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REFERENCES

- [1] A.K. Sinha, Physical metallurgy handbook, The McGraw-Hill Companies, Inc., 2003.
- [2] P. Bała, J. Pacyna, The kinetics of phase transformations during tempering of the new hot working tool steel designed for a large size forging dies. Steel Research International Special edition Metal forming **2**, 407-413 (2008).
- [3] M. Madaj, Copper infiltrated high speed steel based composites with iron additions. Archives of Metallurgy and Materials **54**, 4, 1083-1091 (2009).
- [4] R.W.K. Honeycombe, H.K.D.H. Bhadeshia, Steels. Microstructure and properties, 2nd ed. London: Edward Arnold, 1995.
- [5] P. Bała, The kinetics of phase transformations during tempering of tool steels with different carbon content, Archives of Metallurgy and Materials **54**, 2, 491-498 (2009).
- [6] J.R. Davies, Metallurgy, Processing and Properties of Superalloys, ASM Speciality Handbook: Heat Resistant Materials, ASM International, 1997.
- [7] R.F. Decker, The evolution of wrought age-hardenable superalloys, JOM 32-36 (2006).
- [8] M.J. Donachie, S.J. Donachie, Superalloys. A technical guide, ASM International, Materials Park OH, second edition, 2008.
- [9] M. Durand-Charré, The microstructure of superalloys, CRC Press, 1997.
- [10] Y. Birol, Thermal fatigue testing of Inconel 617 and Stellite 6 alloys as potential tooling materials for thixo-forming of steels, Materials Science and Engineering A **527**, 1938-1945 (2010).
- [11] C. Stöcker, M. Zimmermann, H.-J. Christ, Z.-L. Zhanb, C. Cornet, L.G. Zhao, M.C. Hardy, J. Tong, Microstructural characterisation and constitutive behaviour of alloy RR1000 under fatigue and creep-fatigue loading conditions, Materials Science and Engineering A **518**, 27-34 (2009).
- [12] M. Koori, M. Morishita, K. Yoshikawa, O. Tsuda, Nickel-based heat-resistant alloy for dies. European Patent Application EP0460678.
- [13] P. Bała, Microstructural characterization of the new tool Ni-based alloy with high carbon and chromium content. Archives of Metallurgy and Materials **55**, 4, 1053-1059 (2010).
- [14] P. Bała, New tool materials based on Ni alloys strengthened by inter-metallic phase, Material Engineering (Inżynieria Materiałowa) **3**, 633-636 (2010).
- [15] P. Bała, Microstructural characterization of new tool Ni-based alloy with high carbon content, Archives of Materials Science and Engineering **42**, 1, 5-12 (2010).

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