Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 49
ISSUE 2
December
2011

Microstructure characterization of Ni-Ta-Al alloy with high carbon content

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Received 08.10.2011; published in revised form 01.12.2011

Materials

ABSTRACT

Purpose: The Ni-Ta-Al alloys with high carbon content, strengthened by intermetallic phases, designed for application in high temperatures is presented in the hereby paper. The proposed chemical composition and the results of microstructure investigations as well as hardness in as-cast and after heat treatment condition - are given.
Design/methodology/approach: A test melt of a mass of approximately 1 kg was made in a vacuum furnace, and cast into a ceramic mould. The microstructure of the investigated material was examined by a light microscope Axiovert 200 MAT and the scanning electron microscope FIB Zeiss NEON 40EsB CrossBeam. The heat treatment was performed using the DT 1000 dilatometer made by Adamel Lhomargy, the French Company.
Findings: The main constituents of the microstructure of the Ni-Ta-Al investigated alloy are: the γ phase (matrix), the γ' phase (fine globular precipitates) and as well as primary Ta carbides of MC type and graphite. Primary carbides of irregular shapes are uniformly distributed and not forming agglomerates.
Research limitations/implications: Identification of microstructure components on Ni-Ta-Al with high carbon content materials strengthened by intermetallic phases.
Practical implications: The new model alloy which allows to design a new material for high temperatures applications.
Originality/value: The new chemical compositions and microstructure of high temperature application Ni based materials with high carbon content. Additionally the new alloy, except high carbon volume fraction, is strengthened by intermetallic phases.
Keywords: Tool materials; Ni-based alloys; Intermetallic phases; Carbides

Reference to this paper should be given in the following way:
P. Bała, Microstructure characterization of Ni-Ta-Al alloy with high carbon content, Journal of Achievements in Materials and Manufacturing Engineering 49/2 (2011) 142-149.

1. Introduction

An increased interest of the machine elements production from advanced materials, such as high strength alloy steels, titanium, aluminum or magnesium alloys, forces the development of tool materials for their treatment, especially for forming at high temperatures. Many times, at high temperature, where a variety of attack mechanisms are possible, corrosion resistance may be required. Also, resistance to mechanical damage by many mechanisms such as thermal fatigue or wear is required.

Hot working 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 toughening, 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 and 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. Those 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 range in which those alloys can operate is 600-750°C only.

A development of high temperature creep-resisting nickel-based alloys was mainly made by 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 compounds $Ni_3(Ti, Al)$ designated as γ' [9,10]. The Ni based alloys (named superalloys) are naturally protected to certain extent by the formations of impermeable stable oxides, such as Cr_2O_3 or Al_2O_3 [11].

Several alloys were developed on the concept of Ni-based matrix strengthened by the γ' phase, among others, the alloys of an increased carbon content and a complex chemical composition [12-22].

There are known applications of Ni-based superalloys such as IN617, RR1000 [23,24] or alloys of a complex composition [25] for tools operating at high temperatures. However, a carbon content in such alloys is low (up to 0.1%) and as a result it is not possible to obtain a large fraction of a carbides which would allow to achieve good tribological properties of tools.

Tool materials for operations in higher temperatures based on the matrix of intermetallic phase, called NICRALC alloys, are also noteworthy. Those are Ni-Al-Cr-C alloys and their chemical composition is designated in such a way as to have the matrix constituted solely of the γ' phase [26]. However, obtaining the exactly determined chemical composition and observing strictly the crystallisation procedures, which provide the γ' phase as the matrix, can be difficult under actual industrial conditions.

The Ni-Ta-Al alloy with high carbon content, strengthened by intermetallic phases intended for application at high temperatures, is presented in this paper. The main aim of the research was to determine the microstructural constituents of this, newly designed material on the base of Ni matrix strengthened by intermetallic phase.

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.

The measurements of hardness were carried out with the Vickers HPO250 apparatus.

The heat treatment was performed using the DT 1000 dilatometer made by Adamel, the French Company. Tests were carried out on samples of a size: ∅2x12 mm. The sample was heated at rate of 0.08°C/s to a temperature of 1200°C and cooled at rate of 0.33°C/s to a room temperature.

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

3. Material for investigations

The chemical composition of investigated alloy (table 1) was designed in such a way as to obtain the matrix strengthening by precipitations of 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 an 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 γ' phase together with Al and Ni. Zirconium was added to harden grain boundaries while cobalt to strengthen the γ solid solution and to obtain the microstructure stabilisation. The Ni matrix was chosen due to the lack of its allotropic transformation, which could destabilise the microstructure and properties during a hot-working exploitation.

Table 1.
The chemical composition (wt. %) of the investigated alloy

C	Ta	Al	Zr	P max	S max	Ni
0.81	6.0	3.0	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 of microstructures were made on polished sections parallel and perpendicular to the casting surface.

Fig. 1. View of the investigated alloy casting

4. Results and discussion

The microstructures of the investigated alloy in as-cast state are shown in Figure 2. Large grains, characteristic for as-cast state material, are visible. Dendritic areas are exhibited inside these grains. Primary dendrites with secondary branches are present. Tantalum carbides of MC type are distributed in interdendritic areas (Fig. 2c). Carbides were identified by the EDS analysis (Fig. 3).

The volume fraction of carbides, which is equal 18.4%, was estimated by the point-count method. Primary carbides of irregular oblong shapes and various sizes (Fig. 2) are distributed uniformly not forming aggregates. In addition, a small amount of graphite (about 1.6%) was found (Figs. 2a and d). Probably, the graphite appearance in the investigated alloy microstructures, will cause instability of microstructures at high temperatures exploration.

Hardness measurements were carried out on samples taken from different places on the ingot cross-section. Hardness measured at the ingot surface equals 160 HV and increases in the direction of the casting axis to 170 HV. This is a result of alloying elements segregation in front of the crystallisation, however hardness differences are not large.

Fine precipitates of the intermetallic phase in the γ matrix are seen in the photograph from the scanning electron microscopy (Fig. 2f). This intermetallic phase is so small that its identification was not possible by the EDS analysis. The phase is reach in nickel, aluminium, tantalum. The most probably it is the γ‘ phase (Ni_3AlTa). However, it requires confirmation by means of the TEM. It can be stated that an alloy of a good purity - it means without sulphides and eutectic γ/γ' areas characteristic for nickel-based alloys in as-cast condition - was formed. Such eutectic due to its brittleness is an undesirable component.

The γ phase (Ni based solid solution) constitutes the matrix. The estimation of the γ' phase fraction in as-cast state is very difficult. According to [10,11] tantalum significantly diffuses into the γ' phase causing an increase of this fraction in the alloy. Strengthening of the γ' phase by tantalum is caused by large sizes of its atoms. However, on the basis of the presented hereby results, it seems that at a high carbon content (0.81 %) tantalum prefers the carbide phase. Aluminum content in the investigated alloy is too small to individual formation of γ' phase only with Nickel. By this chemical composition it is more possible to create solid solution than intermetallic phases. Therefore the γ' phase fraction in the investigated alloy is very small. gtrf

The investigated alloy was designed for application at high temperatures. One of the most important problems in Ni-based alloy with high carbon content is carbides stability. Microstructures of investigated alloy after heating and cooling procedures in dilatometer are seen in Figures 4 and 5. As can be noticed (Figs. 4a and b) regardless of applying a slow heating (0.08°C/s) up to a temperature of 1200°C and a slow cooling (0.33°C/s) residues of the primary microstructure (after crystallisation) can be seen in the microstructure. Primary tantalum carbides of MC type remained in the microstructure, all the same their solution process was initiated at a temperature of 1200°C. The presence of the intermetallic phase, which was seen directly after casting, was not found in the microstructure. Heating up to 1200°C caused its dissolutions. On the bases of these two, given above, information it can be stated, that such selection of the temperature from which the investigated alloy will be hyperquenched - in order to remain the stable primary tantalum carbides (of MC type) and to dissolve the intermetallic phase (or phases) - is possible. This provides the possibility of modifying alloy properties by further heat treatments such as stabilisation and aging. On account of the expected applications of the investigated alloy it can be possible to simplify the heat treatment and limit it to two operations: hyperquenching and aging. The slow cooling (0.33°C/s) after heating to 1200°C was chosen in order to determine inclinations to secondary precipitates in the investigated alloy. The secondary tantalum carbides (MC type) precipitated during cooling from 1200°C are shown in Figures 5b and d. Those carbides precipitate in dendritic zones. This indicates the necessity of application the higher cooling rate during hyperquenching. The secondary tantalum carbides are very fine and of irregular shapes. Secondary carbides were identified by the EDS analysis (Fig. 6, tantalum carbides of MC type).

However, it seems that at optimising the hyperquenching temperature in such a way as not to dissolve the primary tantalum carbides (while to dissolve the intermetallic phase) and to increase the cooling rate, the intensity of these effects should be limited. Nevertheless the problem of the carbides instability during aging (precipitation of carbides) still remains. Thus, the temperature and time of aging should be selected in such a way as either to bind total carbon into carbides or to modify the chemical composition by additions of other elements forming stable primary carbides. On account of a graphite presence in the microstructure a much better solution seems to be the chemical composition modification. In addition, hardness in as-cast condition is at low level. Probably the next heat treatment (hyperquenching and ageing), by small volume fraction of γ' phase, could not increase higher hardness.

Further studies on Ni-Ta-Al alloy with a high carbon content will be carried out in order to determine their tribological properties at an ambient temperature as well as at the expected temperature range of operations, eventual forging possibilities, optimization of their chemical composition and heat treatment. The modification of the chemical composition will consist of introducing such elements increasing creep-resistance as chromium and cobalt.

5. Conclusions

The main microstructure components of the investigated Ni-Ta-Al alloy with high carbon content are: the γ phase, which constitutes a matrix, the γ' phase, which occurs as fine globular precipitates, the primary Ta carbides of MC type and graphite. The primary carbides of irregular shapes are distributed uniformly and not forming agglomerates. The alloy has a good purity, it means it is without sulphides and zones of the γ/γ' eutectic characteristic for nickel-based alloys in as-cast condition.

By the high carbon content of investigated alloy chemical composition it seems to that, tantalum prefers the carbide phase. Aluminum content is too small to individual creation of γ' phase with nickel. By this chemical composition it is more possible to create solid solution than intermetallic phases. Therefore the γ' phase fraction in the investigated alloy is very small.

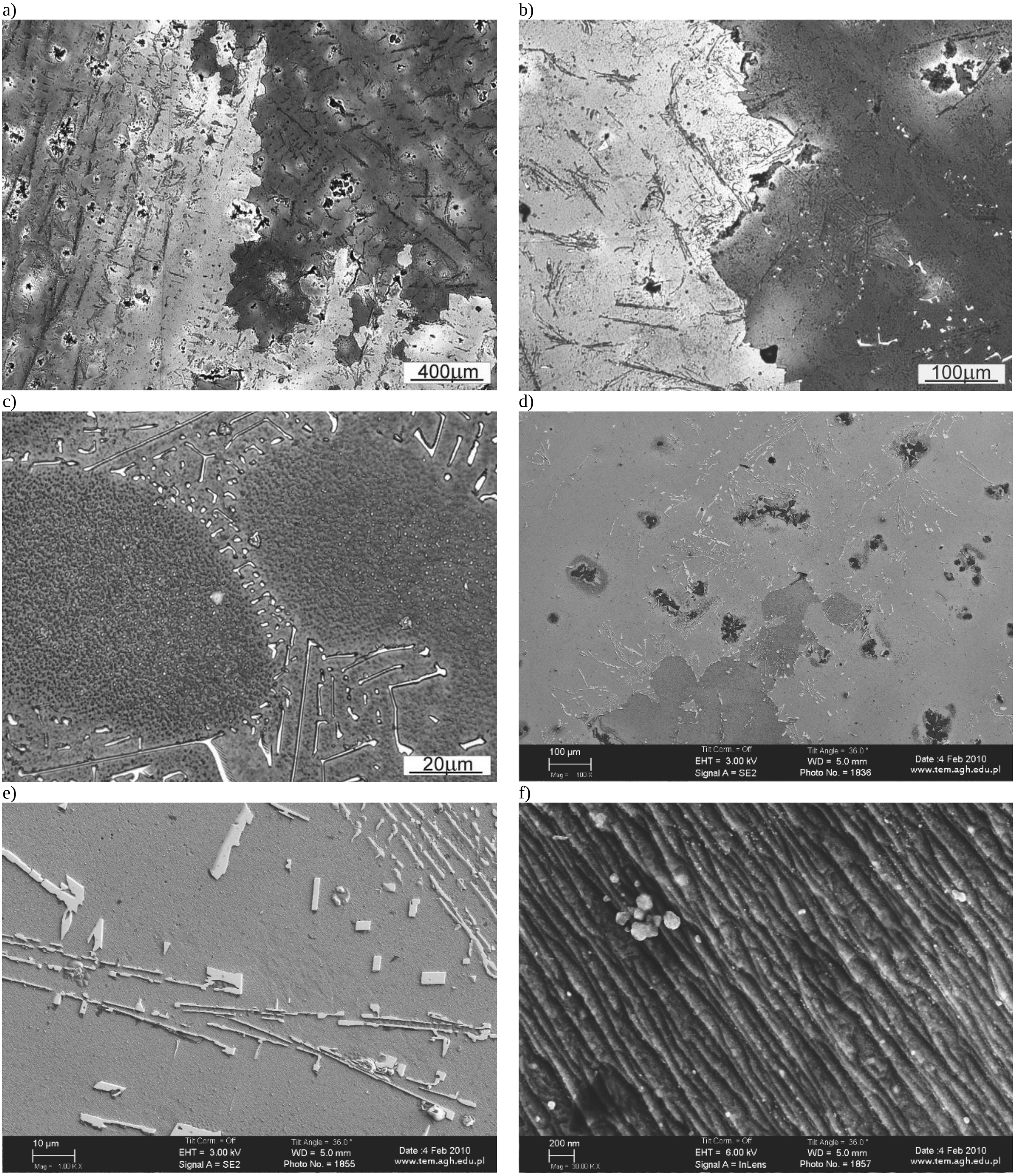
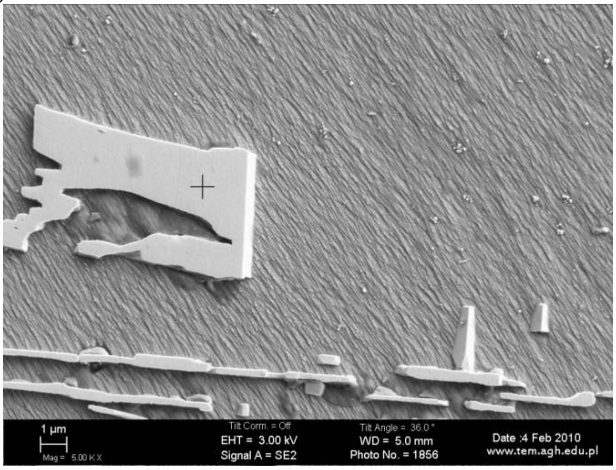


Fig. 2. Microstructure of the investigated alloy. a,b) Area of some grains, c) Carbides in interdendritic areas, light microscope, d) Graphite precipitations,e) Carbide phase morphology, f) Morphology of the intermetallic phases, SEM

a)



b)

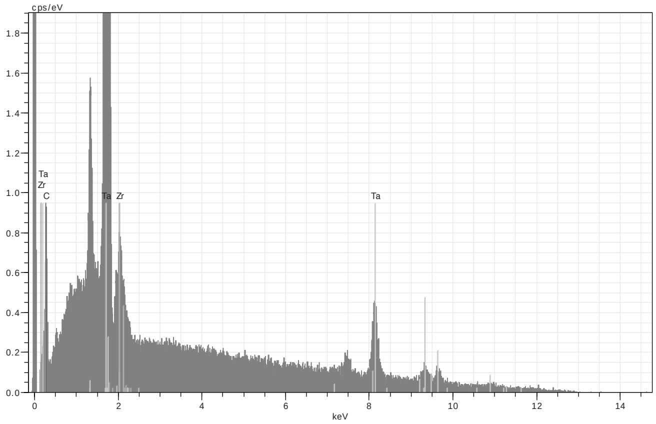
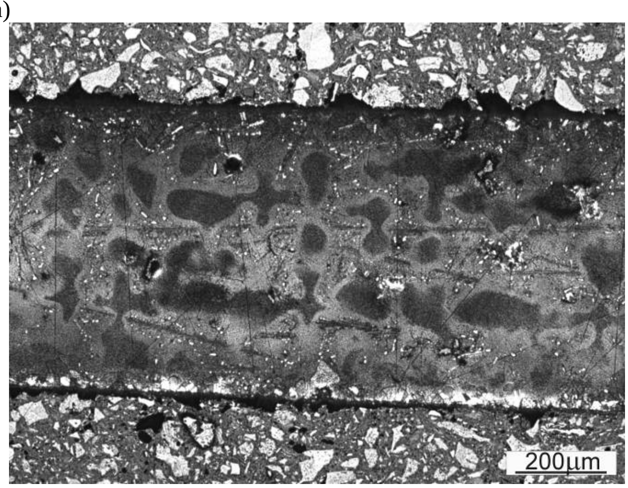
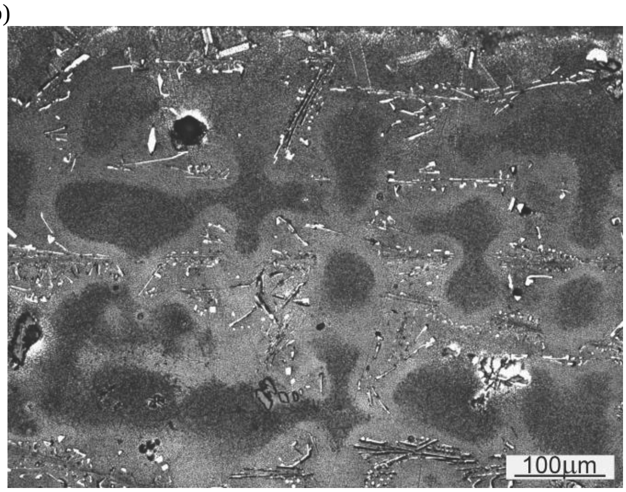


Fig. 3. a) Microstructure of the investigated alloy in as-cast condition with marked zone where from the EDS analysis was performed; b) Characteristic spectrum from marked zone related to TaC carbide

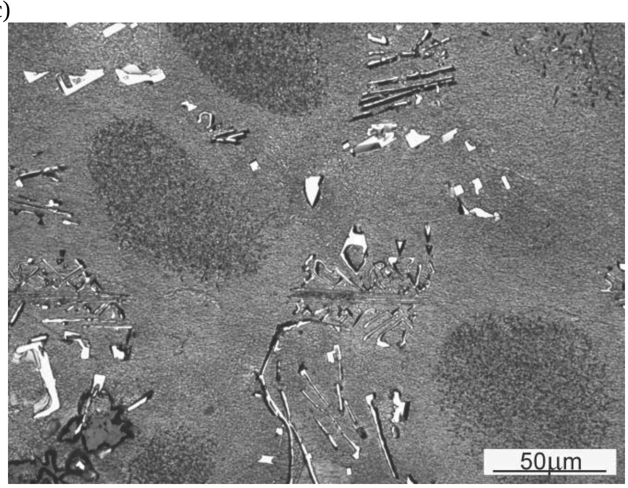
a)



b)



c)



d)

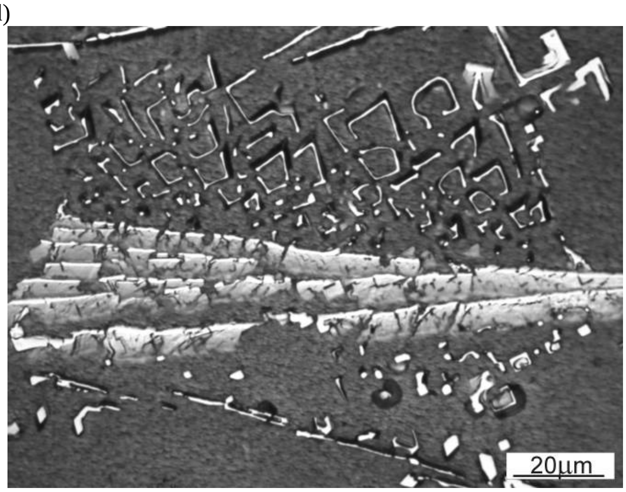


Fig. 4. Microstructure of the investigated alloy after the heat treatment carried out using the dilatometer. a,b) Remains of the primary structure, c,d) Primary tantalum carbides morphology (MC type). Light microscope

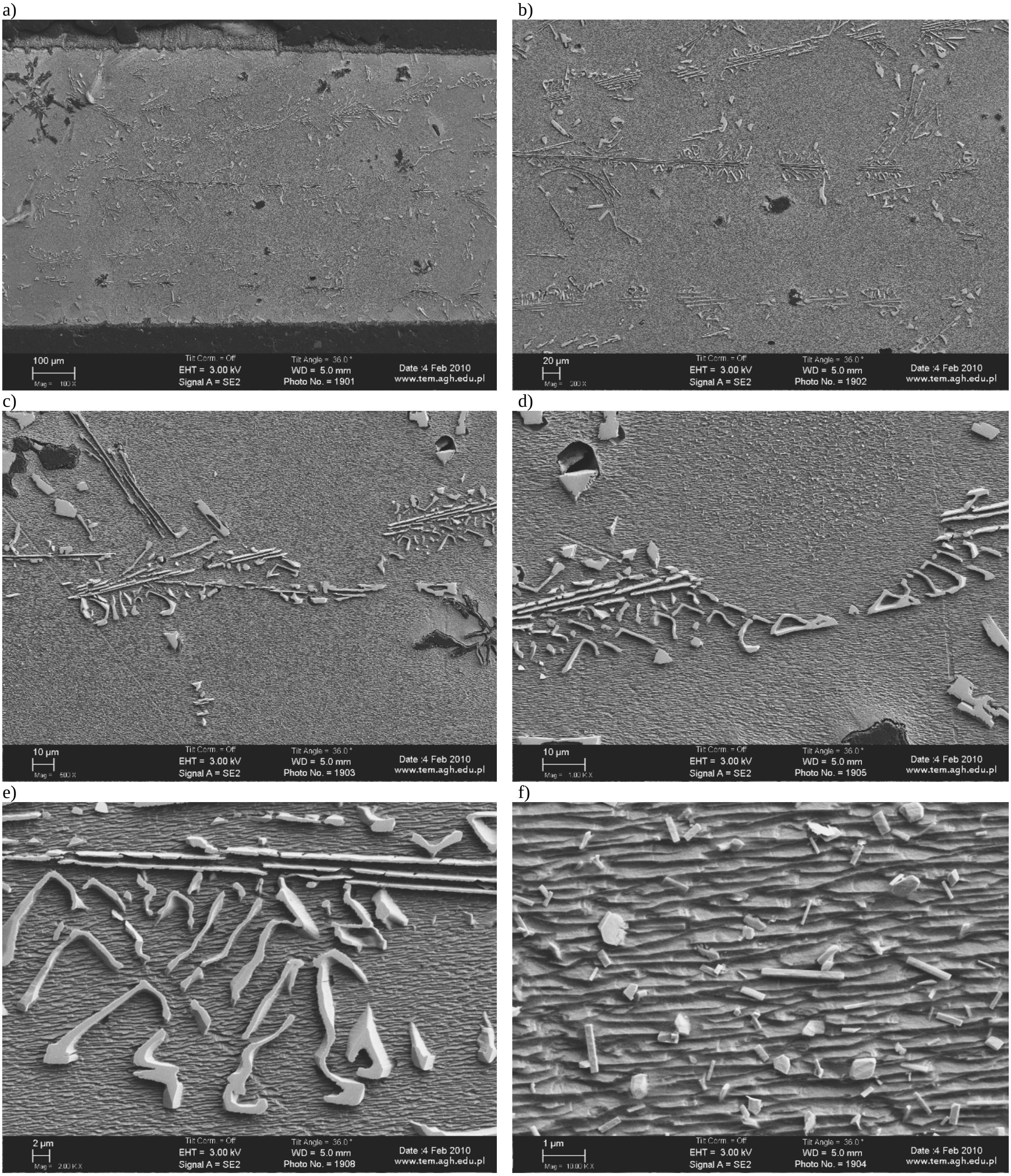
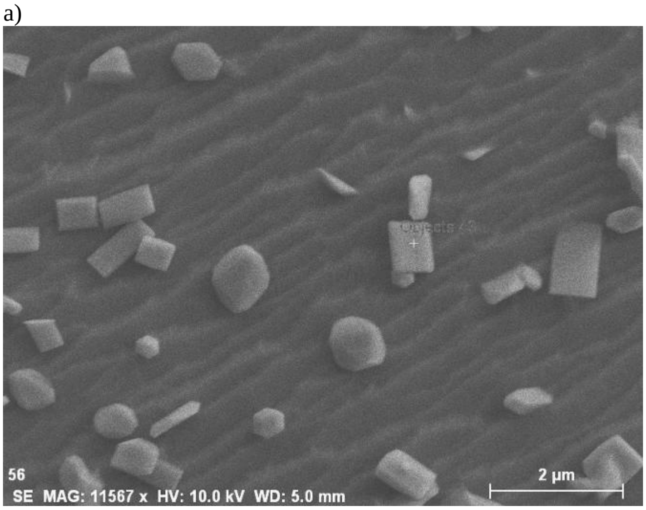


Fig. 5. Microstructure of the investigated alloy after the heat treatment (SEM). a-c) Primary carbides and graphite, d) Primary and secondary tantalum carbides, e) Primary tantalum carbides of MC type, f) Secondary tantalum carbides, precipitated inside dendrites

a)



b)

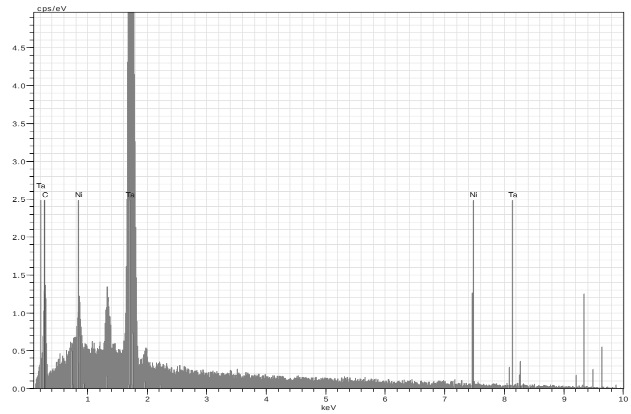


Fig. 6. a) Microstructure of the investigated alloy after the heat treatment with marked zone where from the EDS analysis was performed; b) Characteristic spectrum from marked zone related to TaC carbide

The precipitation of secondary carbides during cooling was found. These carbides are precipitating in dendritic zones. This indicates the necessity to apply higher cooling rates during hyperquenching. The secondary tantalum carbides are very fine and of irregular shapes.

The investigated alloy is the model alloy. A possibility of improving this alloy properties by means of the heat treatment should not be expected at its chemical composition. In the first place the fraction of carbide forming elements should be increased to eliminate graphite, and secondly the fraction of elements forming γ' phase should be increased.

Acknowledgements

The author would like to thank Prof. J. Pacyna and MSc. A. Gruszczyński from AGH University of Science and Technology, Krakow for the valuable help in this research.

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