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Influence of solution heat treatment on the microstructure and hardness of the new Ni-based alloy with a high carbon content

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ABSTRACT

Purpose: The main purpose of the hereby paper was the determination of the temperature and time of solution heat treatment influence on a microstructure and hardness of the newly designed model alloy, being the model Ni-based tool material - intended for operations at high temperatures - strengthened by compositions of intermetallic phases, of a high carbon and cobalt content.

Design/methodology/approach: A test melt of a mass of approximately 1 kg was done in a vacuum furnace, and cast into a ceramic mould. The microstructure of the investigated material was examined by means of the light microscope Axiovert 200 MAT and the scanning electron microscope FIB Zeiss NEON 40EsB CrossBeam. Samples for investigations were solution heat treated from the temperature range: 1020-1170°C. After heating to the desired temperature they were hold at this temperature for 0.5 or for 2 hours and then water-cooled. The volume fraction of primary tantalum carbides and graphite was estimated by the point-count method. Approximately 30 measurements were performed for each variant of the heat treatment.

Findings: The main constituents of the microstructure of the nickel-base investigated alloy in as-cast state are: the γ phase, which constitutes the matrix, the γ' phase (γ' phase occurs as fine globular precipitates) as well as primary Ta carbides of MC type. Primary carbides of irregular shapes are uniformly distributed not forming agglomerates. Solution heat treatment of the investigated alloy from higher and higher temperatures causes a slow increase of a graphite fraction, nevertheless an application of a temperature of 1170°C significantly increases its fraction.

Research limitations/implications: The investigated alloy is the model alloy. Taking into account its chemical composition the possibility of improving its properties by means of the heat treatment should not be expected. 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 to obtain the possibility of the modification of properties by means of solution heat treatment and aging. Nonetheless the achieved results will be utilised at the chemical composition modification of Ni-based alloys of a high carbon content.

Practical implications: Broadening the knowledge on the carbides stability in Ni-based alloys, which will help in designing new Ni-based alloys of a high carbon content.

Originality/value: Determination of the stability of primary carbides at high temperatures in Ni-based alloys of a high carbon and cobalt content.

Keywords: Tool materials; Ni-based alloys; Intermetallic phases; Carbides

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MATERIALS

1. Introduction

An intensive development of several industry branches in the last several dozen years, introduced more sophisticated requirements for technological processes and products. Very often materials used in production or components of final products have to operate at higher temperatures, at higher loads and in corrosive conditions. This situation forces the development of tool materials, which have to satisfy steadily increasing requirements.

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 treatment, 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 [1-4].

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 [5]. 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 [6].

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 Ni_3(Ti, Al) designated as γ' [7,8].

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 [9-16].

There are known applications of Ni-based superalloys such as IN617, RR1000 [17,18] or alloys of a complex composition [19] for tools operating at high temperatures. However, a carbon content in such alloys is low (not exceeding 0.1%) and as a result obtaining a large fraction of a carbide phase - which would allow to achieve good tribological properties of tools - is not possible.

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 selected in such a way as to have the matrix constituted solely of the γ' phase [20]. 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 main purpose of the hereby paper was the determination of the influence of a temperature and time of solution heat treatment on the microstructure and hardness of the newly designed model Ni-based alloy strengthened by intermetallic phases of a high carbon and cobalt content, intended for operations at high temperatures.

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 hardness measurements were performed with the Vickers HPO250 apparatus.

The carbon content was measured by means of the LECO CS-125 analyser.

Samples for examinations were solution heat treated from the temperature range: 1020-1170°C. After heating to the needed temperature they were hold either for 0.5 or for 2 hours and then water-cooled. The volume fraction of primary carbides of tantalum and graphite was estimated by the point-count method (app. 30 measurements were performed for each heat treatment variant).

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 the 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 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	Co	P max	S max	Ni
0.88	6.0	3.0	0.2	20.0	0.01	0.01	Bal.

A test melt of a mass of approximately 1 kg was done in a vacuum furnace, and cast into a ceramic mould. The casting together with the first cut sample (as an example) is shown in Fig. 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. Research results and discussion

The microstructure of the investigated alloy, after casting, was described in detail in reference [21]. For carrying the discussion of the obtained results only the most important features of the microstructure will be presented here. The microstructures of the investigated alloy in as-cast state are shown in Figures 2 and 3. Large grains, characteristic for as-cast state material, are clearly visible (Fig. 2a). Dendritic areas are exhibited inside these grains (Fig. 2b). Primary dendrites with secondary branches are seen. Tantalum carbides of MC type are distributed in interdendritic areas (Figs. 2c and 3a,b). In addition, a small amount of graphite was found (Figs. 2a,b and 3c). The presence of intermetallic phase γ' was also found (Fig. 3d). The volume fraction of carbides and graphite was estimated by the point-count method. The volume fraction of carbides equals 23.8%, and graphite equals 1.0%. Primary carbides of irregular, oblong shapes and various sizes (Fig. 2c) are distributed uniformly, not forming aggregates.

Hardness measurements were carried out on samples taken from different places on the ingot cross-section. The hardness measured at the ingot surface equals 222 HV and increases in the direction of the casting axis to 243 HV. This is a result of alloying elements segregation in front of the crystallisation, however hardness differences are not large.

It should be mentioned, that the investigated material was not processed by forging, which could cause small differences in properties and in microstructures. Microstructures of the investigated alloy after solution heat treatment from the temperature range 1020-1170°C (time: 30 minutes or 2 hours, water-cooling) are presented in Figs. 4 and 5. In relation to the as-cast state (Fig. 2) the dendritic structure was not revealed. However, these spaces can be distinguished due to the carbide phase shape, since not dissolved tantalum carbides are seen in the microstructure (nevertheless their fraction decreased). Dendritic ranges are slightly growing. At each increase of the solution heat treatment temperature the graphite fraction increased while the primary tantalum carbides fraction decreased.

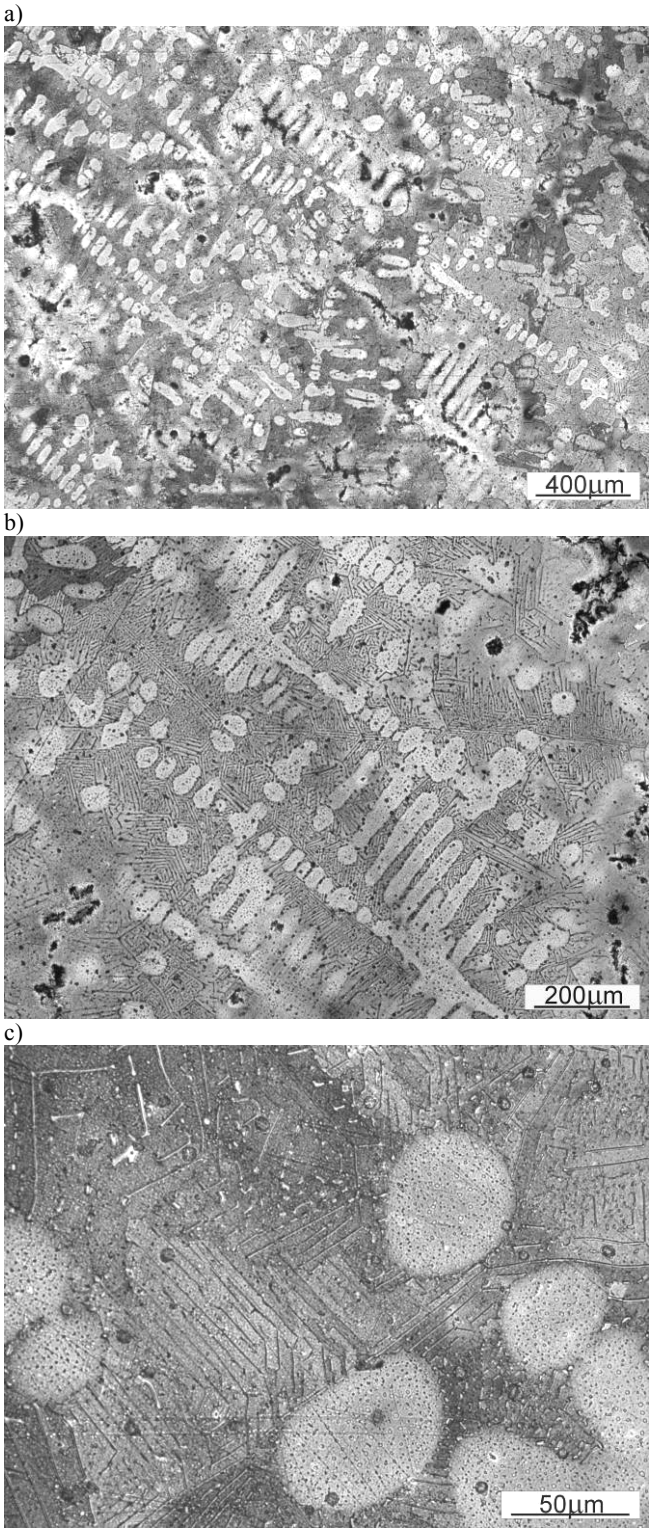

Fig. 2. Microstructure of the investigated alloy. a) Area of some grains, b) Dendrites within a single grain, c) Carbides in interdendritic areas. The light microscope

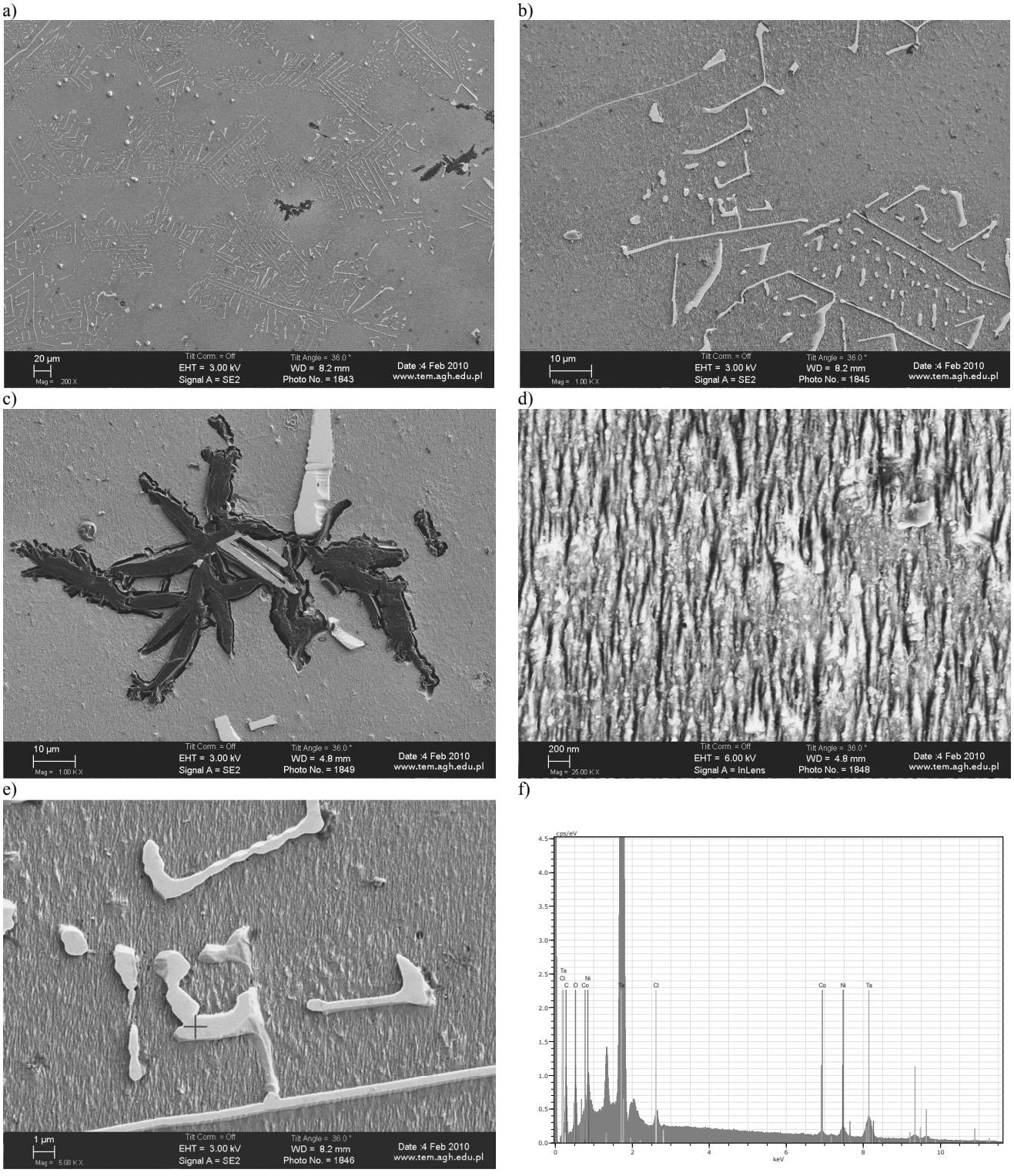

Fig. 3. Microstructure of the investigated alloy an as-cast state. a,b) Carbide phase morphology; c) Graphite precipitations; d) Morphology of intermetallic phase; e) Carbides with marked zones where from the EDS analysis was performed, SEM; f) Characteristic spectrum from marked zone

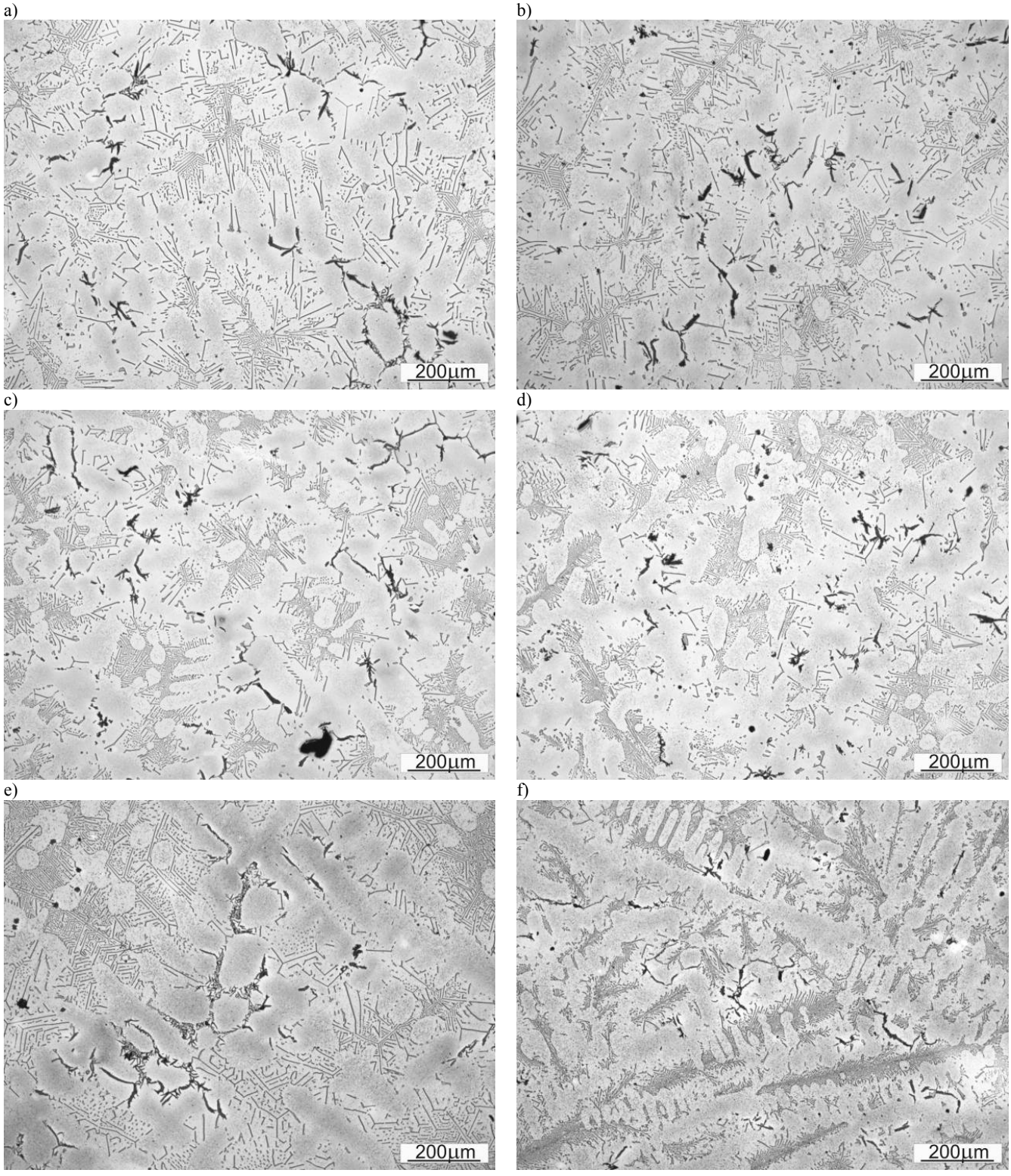

Fig. 4. Microstructure of the investigated alloy after solution heat treatment in water from a temperature: a,b) 1020°C; c,d) 1050°C; e,f) 1080°C. a,c,e) heating time at each temperature 0.5 h; b,d,f) heating time at each temperature 2 h. Light microscope

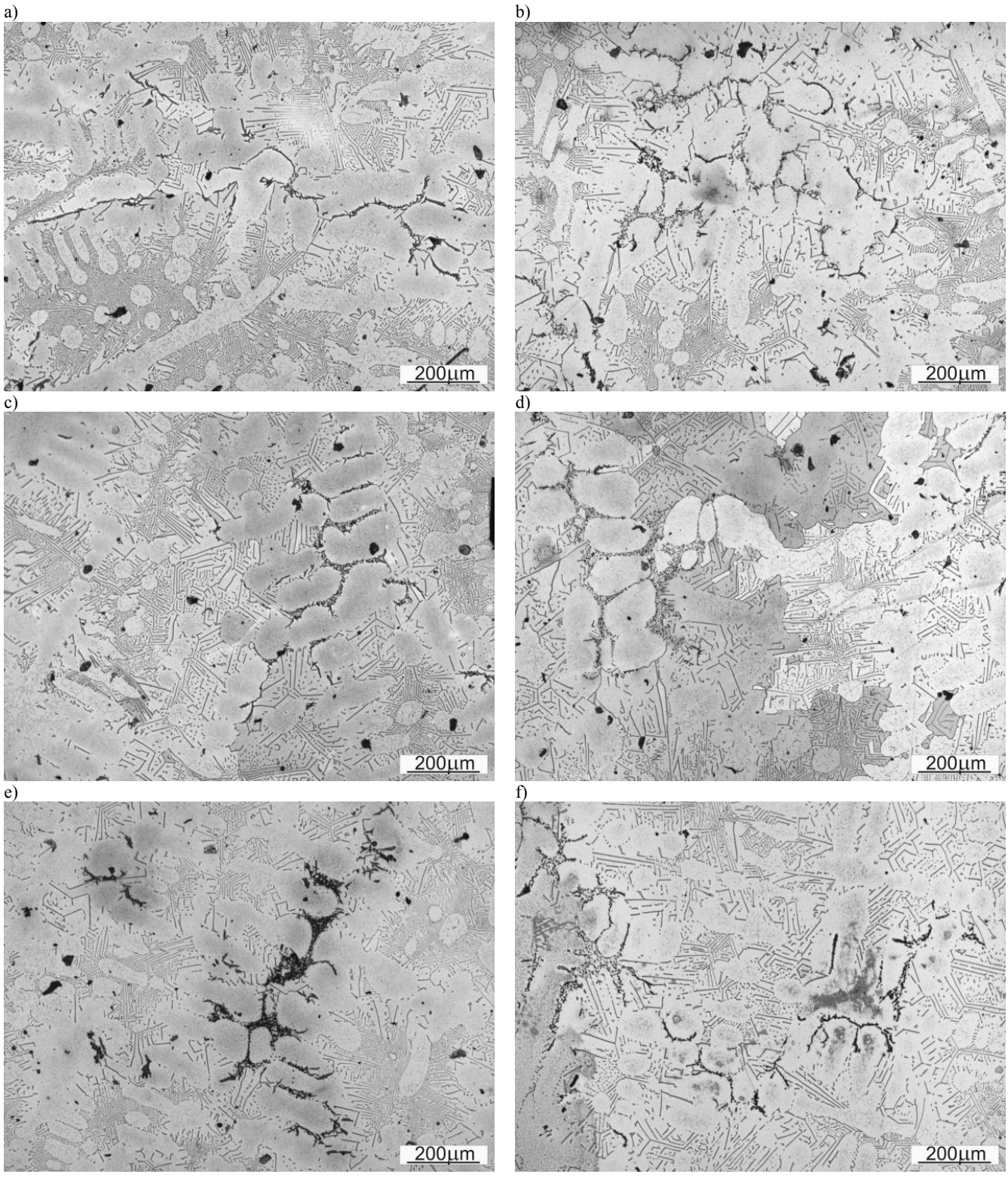

Fig. 5. Microstructure of the investigated alloy after solution heat treatment from a temperature: a,b) 1110°C; c,d) 1140°C; e,f) 1170°C. a,c,e) heating time at each temperature 0.5 h; b,d,f) heating time at each temperature 2 h. Light microscope

It was found that up to a temperature of 1050°C the carbide decomposition into graphite occurred mostly on grain boundaries. From a temperature of 1080°C graphite was formed also inside grains.

On the basis of the microstructure photographs shown in Figs. 4 and 5 the analysis of changes in the volume fraction of primary tantalum carbides and graphite was performed. 10 photos were taken for each analyzed solution heat treatment variant.

The influence of a temperature and solution heat treatment time on the volume fraction of primary tantalum carbides and graphite is illustrated in Fig. 6. As it can be seen, the graphite fraction very slowly increases with the temperature increase, nevertheless barely the application of 1170°C causes a strong increase of its fraction. It should be mentioned that the investigated alloy is inclined for graphitization. The observation that the heating time increase from 30 minutes to 2 hours does not influence the primary carbides and graphite fractions seems surprising. The deciding role belongs to the temperature only. Probably in the moment of carbide decomposing into graphite a large tantalum concentration locally occurs. It may be, that a high temperature is needed for the initiation of the carbide decomposition which can not further proceed due to the tantalum diffusion rendered difficult by cobalt atoms, which - in turn - slows down the process. That is why the primary tantalum carbides fraction decreases with the temperature increase, though it occurs abruptly. Up to a temperature of 1080°C the carbide fraction stays on a similar level (app. 16%) followed by a drop to app. 10%. However even heating to 1170°C does not cause a significant decrease of its fraction. An influence of a temperature and solution heat treatment time on the hardness of the investigated alloy is presented in Fig. 7.

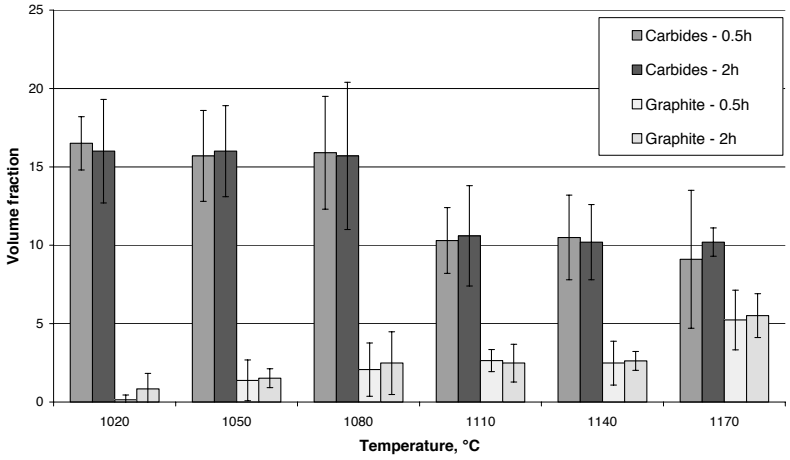

Fig. 6. The influence of temperature and time of solution heat treatment on the volume fraction of tantalum carbides and graphite

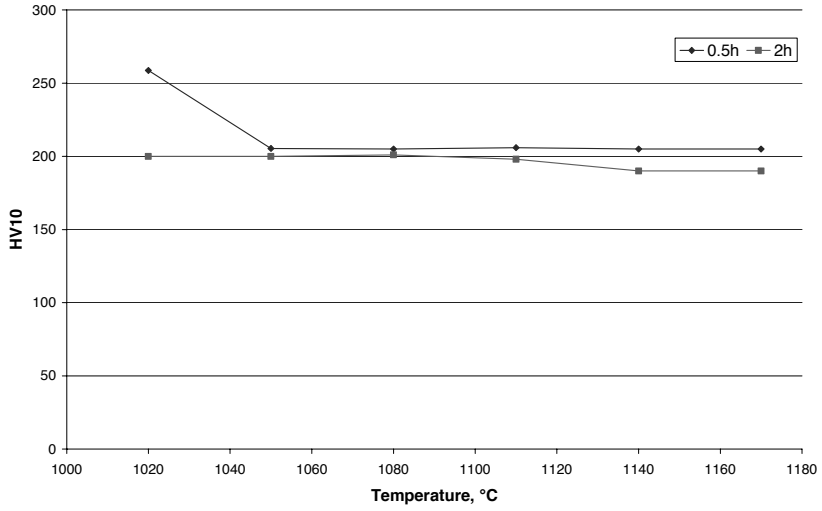

Fig. 7. The influence of a temperature and time of solution heat treatment on the hardness of the investigated alloy

As can be seen the hardness remains on a similar level, regardless of the applied temperature and time of solution heat treatment. A small increase for solution heat treatment from 1020°C (time: 30 minutes) can be caused by a precipitation or increase of intermetallic phases, since in spite of a high temperature application (1020°C) time of solution heat treatment was short. Thus, it seems that it would be possible to treat the investigated alloy from the temperature range: 1050-1100°C, applying short holding times. The hardness mainly depends on the matrix and graphite fraction, but it is on too low level in the examined samples to play an essential role.

5. Conclusions

The main microstructure components of the investigated Ni-based alloy with high carbon content in as-cast state are: the γ phase, which constitutes a matrix, the γ' phase, which occurs as fine globular precipitates and the primary Ta carbides of MC type. The primary carbides of irregular shapes are distributed uniformly, not forming agglomerates.

The investigated alloy is prone to graphitisation. Solution heat treatment from higher and higher temperatures causes that the graphite fraction increases slowly, nevertheless only the application of a temperature of 1170°C strongly increases its fraction. Prolongation of a heating time from 30 minutes to 2 hours does not influence the graphite and primary carbides fraction. The deciding role plays only the temperature.

The hardness remains on a similar level, regardless of the applied temperature and time of solution heat treatment. The hardness of the investigated alloy depends on the matrix and graphite fraction, but it is on too low level in the examined samples to play an essential role.

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.

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