Journal of Achievements in Materials and Manufacturing Engineering

VOLUME 31
ISSUE 2
December
2008

Identification of dynamically precipitated phases in hot-working Inconel 718 alloy

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Received 11.09.2008; published in revised form 01.12.2008

Materials

ABSTRACT

Purpose: The main purpose of this paper was to analyze how localized flow/structural inhomogeneties that may develop during hot deformation can affect a precipitated phases and to determine what kind of particles are present in the microstructure of hot-worked Inconel 718 superalloy.

Design/methodology/approach: Compression tests were carried out on precipitations hardenable nickel based superalloy of Inconel 718 at constant true strain rates of 10^{-4} and $4x10^{-4}s^{-1}$ at temperature of 720 and 850°C. The dynamic behaviour were explained through observation of the microstructure using standard optical, scanning and transmission electron microscopy. Precipitated phases were identified using EDS technique and based on selected area diffraction pattern.

Findings: Microstructural observations of deformed at high temperatures, previously solution treated Inconel superalloy revealed non uniform deformation effects. Distribution of molybdenum- and niobium-rich carbides affected by localized flow was found. Microstructural examination of the alloy also shown shear banding penetrating through the whole grains.

Practical implications: The experiments on hot deformation of age hardenable Inconel 718 superalloy and the analysis of dynamic precipitation process have a practical aspect. This interaction could become an important feature of high temperature performance and may also influence the production of specific structures of this material.

Originality/value: Even though the number of research has focused on the hot deformation behaviour of Inconel 718, there is still scarcity of data referring to the analysis of dynamic structural processes which operate during hot deformation of this precipitation hardenable alloy: in particular dynamic precipitation and dynamic particles coarsening.

Keywords: Alloys; Working properties of materials and products; Plastic forming; Dynamic precipitation

1. Introduction

Inconel 718 is a nickel-based superalloy extensively used in fabrication of critical components for turbine because of its excellent mechanical properties at elevated temperatures and good corrosion resistance. Since these components withstand high alternating stresses and creep loads, the suitable microstructure with fine grains must be obtained by controlling forging processes. In the turbine disc application, the standard processing, the high strength processing and the direct age processing have been applied in order to reach the required properties. Although the number of studies has focused on the hot deformation behaviour of Inconel 718, there is still shortage of data referring to the analysis of dynamic structural processes which operate during hot deformation of this precipitation hardenable alloy: in particular dynamic precipitation and dynamic particles coarsening. In presented research work the interaction of strain localization and dynamic precipitation process was tested during hot compression of Inconel 718.

The contribution of flow localization to the strain hardening or flow softening and the flow stress-strain behavior during hot deformation of precipitation hardenable alloys is still a subject of extensive research. The interaction between the flow localization and dynamic precipitation process has been the subject of very limited research [1-13]. There is a scarcity of data which refer to specific features of phase transformation processes in precipitation hardenable alloys. Moreover, the existing data does not allow generalizing structural features of dynamic precipitation and simplifying structural description of the process. The experiments on hot deformation of age hardenable alloys and the analysis of dynamic precipitation process have a practical aspect. This interaction could become an important feature of high temperature performance and may also influence the production of specific structures of material. The present research work contributes some new information about the precipitation process which occurs in solution treated nickel based superalloy deformed at elevated temperatures and was inspired by the studies of previous researches performed on Cu-Ni-Si-Cr-Mg, Cu-Ti and low carbon steel [1-6]. Non-uniform deformation was observed during hot deformation particularly, which was found to have an effect on dynamic structural processes and, subsequently, the microstructure and mechanical properties of the hot deformed alloys. The plastic instability resulted from the interaction of discontinuous precipitation and shear bands development. The flow localization was accompanied by dynamic particles coarsening within shear bands. The flow stress-strain behavior of Cu-Ni-Cr-Si-Mg alloy was affected and controlled by dynamic precipitation and its interaction with the strain localization. Additionally, the dynamic coarsening within shear bands was reported to be responsible for further flow softening during hot compression of the material. The shearing process was found to be a self-induced one, i.e. the flow localization accelerated discontinuous precipitation and the precipitation coarsened within the sheared area, which promoted further flow softening. In order to test the interaction of deformation with the precipitation process, hot compression tests were performed on the wrought nickel based superalloy Inconel 718. In718 alloy was chosen since it is hardenable and therefore suitable for testing continuous precipitation interaction with deformation during hot deformation. Thus, the purpose of the described experiments was to study the structural changes that occur during hot deformation of supersaturated Inconel alloy below the solvus temperature (dynamic precipitation conditions). Emphasis was placed on the determination of phases dynamically precipitated during hot working of the alloy.

The role of carbides in superalloys is often questioned. However, many researchers have noted that carbides in microstructure of nickel based supearalloys must be tolerated since their influence on stability of the matrix, ductility of a material. Therefore, understanding of their desirable morphology and variety is critical to the superalloys as constructional materials [15]. Carbides like geometrically close packed phases such as gamma prime (γ') are dispersed in a face centered cubic (γ) austenitic matrix. Molybdenum, niobium, chromium, vanadium and titaniumin are carbide formers elements and commonly produced MC, $M_{23}C_6$, M_6C or M_7C_3 in superalloys. Their morphology depends on the base metal, alloy composition, heat treatment and deformation parameters. MC type carbides usually are characterized by coarse, random, blocky or cubic or script morphology. $M_{23}C_6$ carbides occur in grain boundaries or on dislocations/stacking faults generally in form of discrete particles or platelets. M_6C carbides precipitate in grain boundaries in blocky or acicular/Widmanstätten intragranular form. M_7C_3 type of carbides takes blocky intragranular morphology (e.g. Cr_7C_3). The γ' phase (Ni_3Al) exists in form of blocky and spherical. However, the γ" phase (body centered tetragonal Ni_3Cb) is characterized by disk or platelet precipitates in form of intergranularly as a cellular (pearlite-like) structure or intragranularly as acicular plates [15].

Table 1.
Chemical composition of Inconel 718 (wt.%)

C	0.0197
P	<0.011
S	0.0064
Cr	17.957
Si	0.0640
Nb	4.5880
Co	0.0527
Zr	0.0159
Ta	0.0221
Ti	0.8734
Fe	16.665
Mo	2.6400
Al	0.4917
Mn	0.0537
V	0.0137
W	0.01322
Ni	balance

2. Material for research

The uniaxial compression tests at different temperatures were performed on solution treated precipitations hardenable nickel based superalloy Inconel 718 (Table 1) in order to investigate the effect of the hardening phases on hot deformation behavior. In order to intensify an interaction of phase transformation and deformation processes, hot compression tests were performed at low constant strain rate within the temperature range corresponding to temperatures of precipitation of hardening phases. Long compression test allows to reach large enough deformation value and long enough deformation time which was required for diffusion controlled process to develop during deformation test. The cubicoidal samples were deformed by means of computerized Gleeble test equipment. The properties were explained through observation of the microstructure using standard optical and advanced transmission and scanning electron microscopy technique. Hitachi 3400 scanning electron microscope equipped with an energy-dispersive spectroscopy (EDS) system operated at 20 kV was used. Specimens for transmission electron microscopy (TEM) were prepared by electrochemical thinning in self-made jet polisher at 20V and -10°C using a solution of 10% perchloric acid and 90% methanol by volume. TEM observations were performed using JEOL 2010 electron microscope operated at 200kV. The chemical information on precipitates was obtained using EDS operated in scanning transmission electron microscope (STEM) mode.

3. Description of achieved results

Hot compression tests were performed on the solution treated precipitation hardenable nickel based superalloy Inconel 718 at 720-850°C with a constant true strain rates of 10^{-4} and $4x10^{-4}s^{-1}$. The characteristic structure of the solution treated Inconel 718 sample deformed at 800°C with a strain rate of 10^{-4} s^{-1} is shown in Fig. 1. Coarse slip bands in the grain interiors and localized flow shear bands were observed.

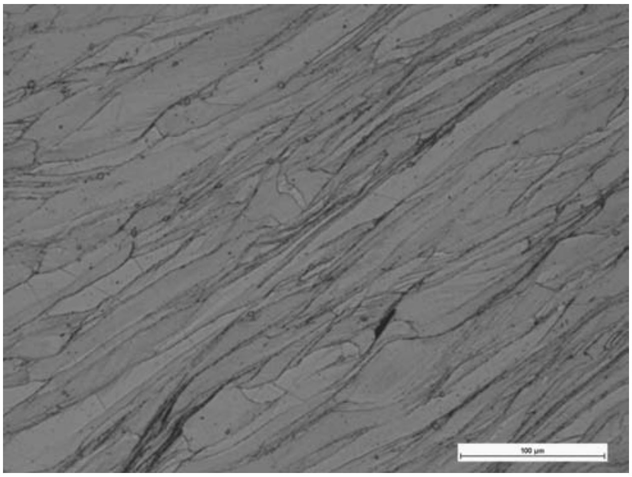


Fig. 1. Micrograph of the alloy deformed at 800°C, strain rate of $10^{-4}s^{-1}$

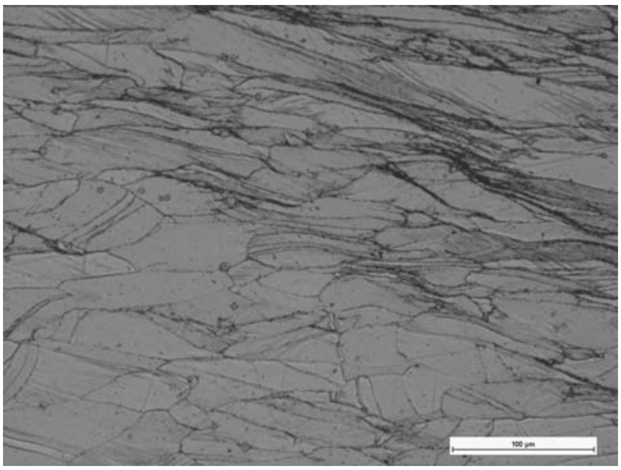


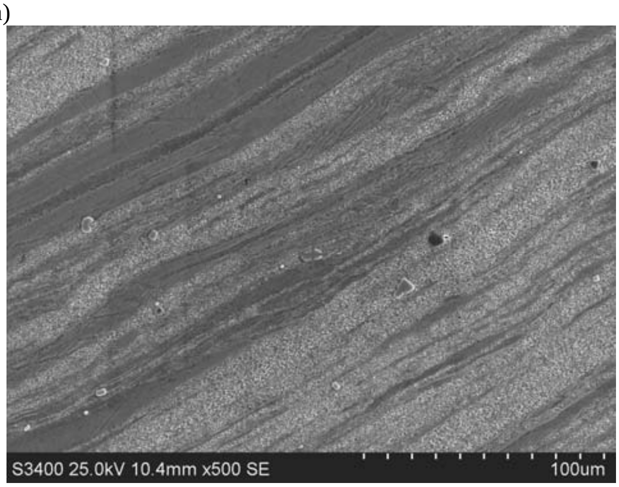
Fig. 2. Optical micrograph for the sample of In718 deformed at 850°C, strain rate of $4x10^{-4}s^{-1}$

Non-uniform deformation and coarse slip bands were also observed in the microstructure of the samples of Inconel deformed at 850°C with strain rate of $4x10^{-4}s^{-1}$ (Fig. 2).

Both figure let us see coarsened particle affected by localized flow. Identification of the particles were provide by scanning electron microscope combined with EDS technique.

Fig. 3a shows similar results, to those observed by optical metallography, by using SEM at higher magnifications, for the solution treated, deformed at 850°C with strain rate of $10^{-4}s^{-1}$. Large particles along the shear band are seen in the deformed solution treated specimen. Microstructure shown in Fig. 3 was chosen for the precipitate identification because it contains typical particles observed with using scanning electron microscope. From Fig. 3, we can see that the precipitates present are rounded or elongated to direction of deformation bands. Fig. 3b shows a typical EDS spectrum of the particle from Fig. 3a (marked with red cross), from which it can be preliminarily concluded that the precipitate contains Mo, which is carbide formers, but contained appreciable amounts of Cr, Fe and Ni in addition to Ti. The presence of Cr, Fe and Ni in the EDS spectrum may be associated with matrix elements, or could be attributed to the nature of the carbides.

a)



b)

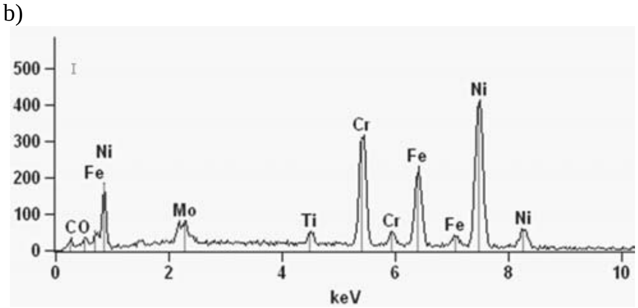


Fig. 3. a) SEM microstructure of the Inconel 718 alloy deformed at 850°C with strain rate of $10^{-4}s^{-1}$, b) EDS spectra of the particle marked with the red cross

The X-ray mapping on the SEM from the microstructure observed at this magnification (Fig. 4) showed that the particles contained principally Mo, which suggested that the examined particle was MoC.

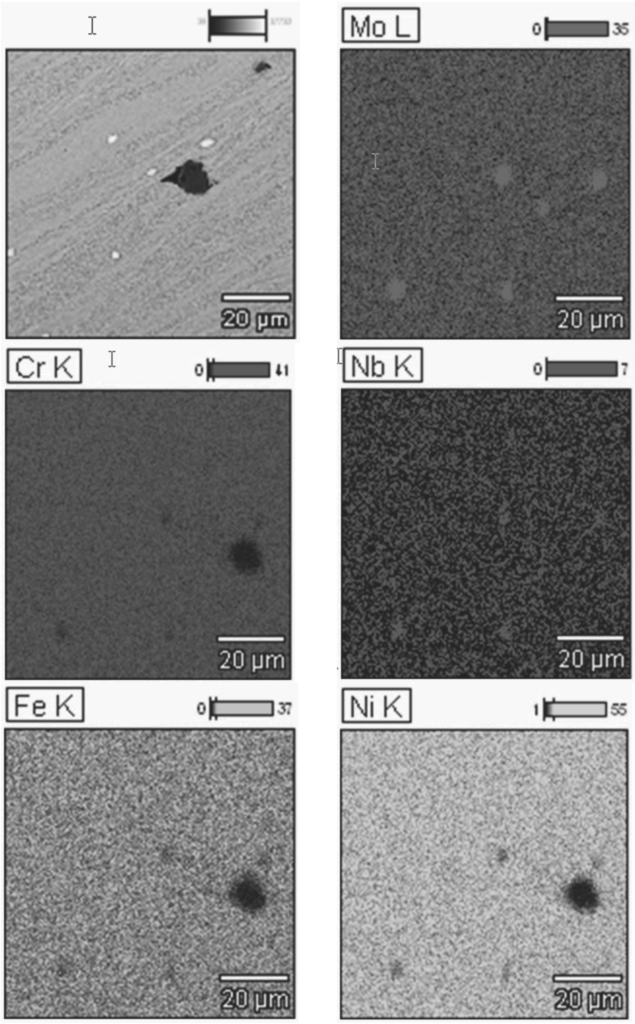


Fig. 4. SEM micrographs of specimens and X-ray mapping results for the precipitates within shear band observed in Inconel 718 alloy deformed at 800°C and strain rate of $10^{-4}s^{-1}$

Further confirmation of the carbides was needed and was carried out by TEM studies. TEM observations of the samples deformed at 720 and 850°C with both strain rates confirmed that localized deformation may promote intensive dynamic precipitation and coarsening of the particles within shear bands (Fig. 5 and 6).

In conformance with the findings from scanning electron microscope SEM, Nb-rich carbides were observed by TEM. These carbides are usually of irregular shape and were most often found within shear bands (Fig. 5).

The carbide is shown in Fig. 7 along with a diffraction pattern from the particle and its surrounding matrix. The particle contained mainly Nb, with a small amount of Ti and Cr. It has a cubic structure with lattice parameter a=0.43nm, which matched well with the Nb-type carbides (a=0.44nm) reported by Wilson [14].

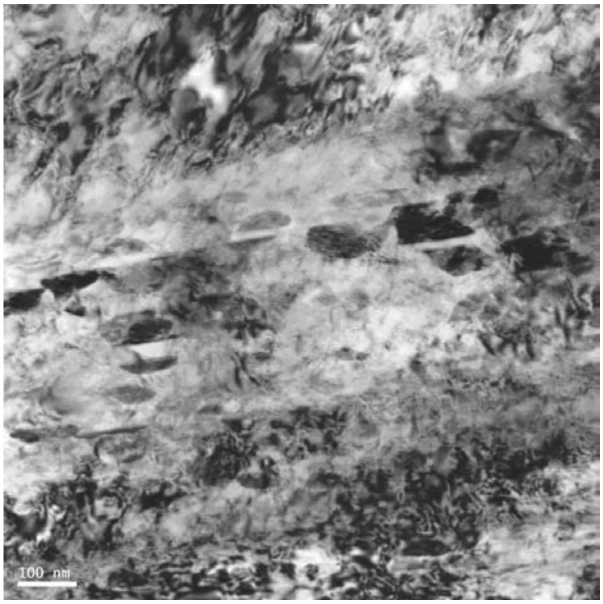


Fig. 5. Microstructure of the alloy subjected to compression test at 720°C, strain rate of $10^{-4}s^{-1}$. Carbides phase precipitation within shear band

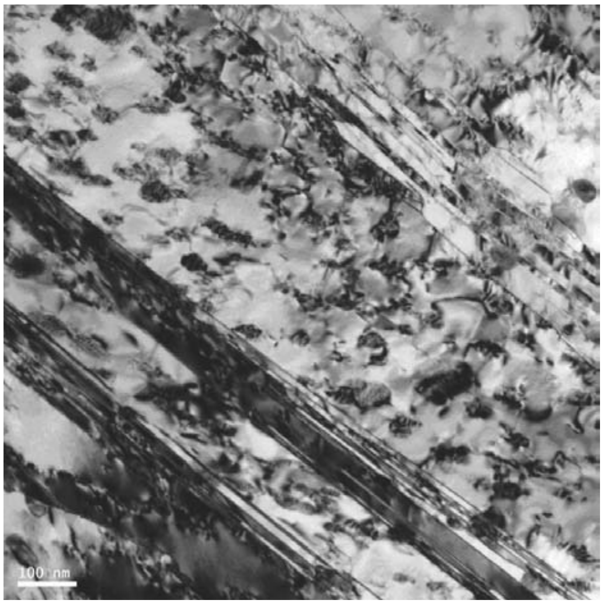


Fig. 6. Microstructure of the alloy subjected to compression test at 850°C, strain rate of $4x10^{-4}s^{-1}$. Carbides phase precipitation within shear band

There is no crystallographic orientation relationship between particle and the matrix. Similar particles were often observed within the localized flow areas of deformed microstructure of the examined Inconel. These examinations indicate that the particles are associated with secondary NbC.

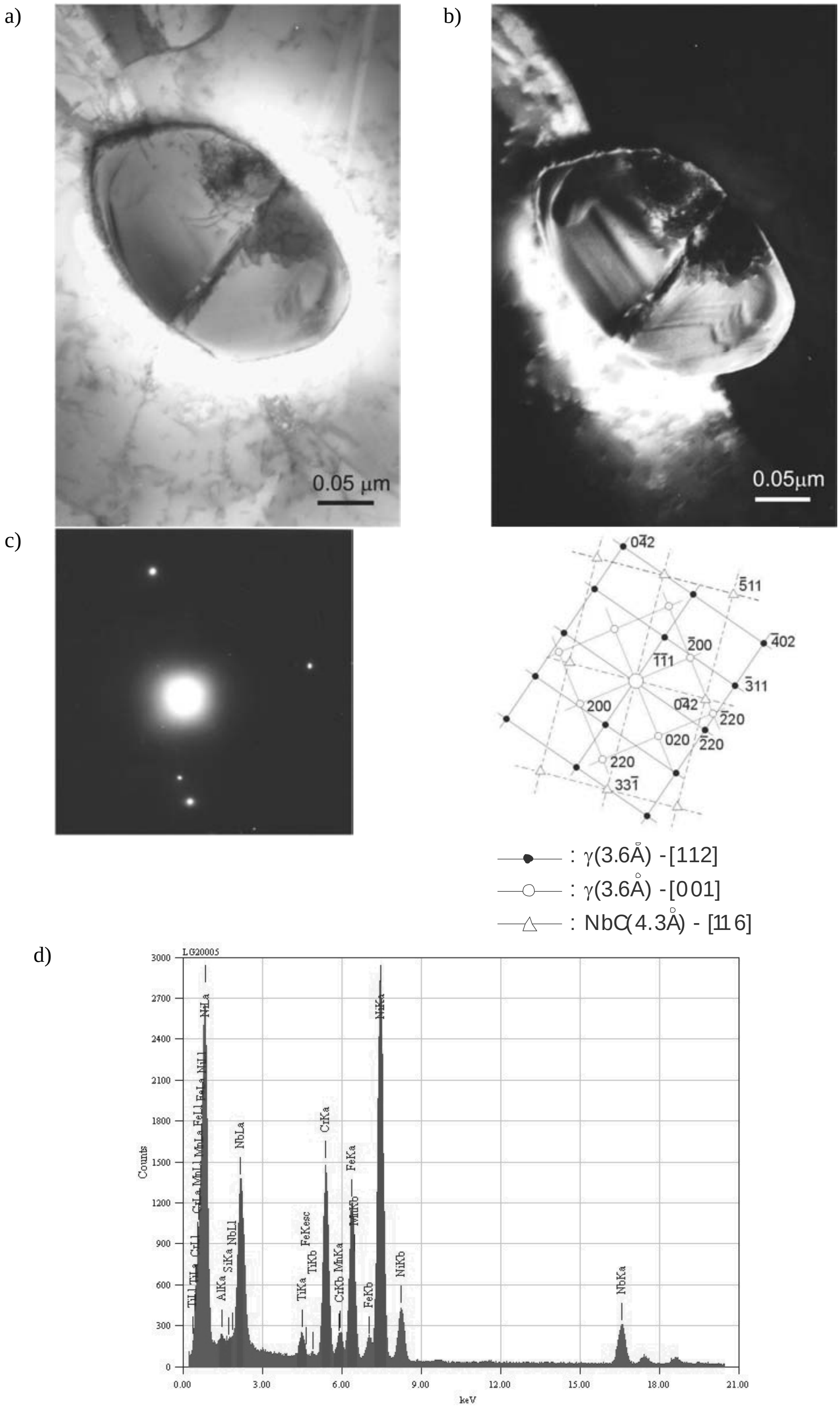


Fig. 7. Nb-rich carbides in the sample of Inconel 718 deformed at 720°C with strain rate of $10^{-4}s^{-1}$; a) TEM bright field, b) TEM dark field, c) indexed diffraction pattern (SADP) – zone axis was [$\bar{1}$ $\bar{1}$ 1], d) EDS spectra of analyzed particle

The crystallographic orientation of the particles appears to be related to that of the matrix on one side of the shear band. Based on the diffraction from the particle and its surrounding matrix, the following matrix-particle relationships were obtained: $(112)\gamma//(116)_{particle}$ and $(001)\ \gamma//(116)_{particle}$. The EDS spectra showed the particles to be rich in Nb, but also contained some amounts of Ti, Cr and Fe.

EDS linear analysis from a typical, large particle showed that this type of particle contained mainly Nb, which indicates that these particles are NbC carbides (Fig. 6e).

4. Conclusions

The dynamic behaviour was explained through observation of the microstructure using standard optical, scanning and transmission electron microscopy.

Structural observations of solution treated Inconel 718 deformed at high temperatures, reveal non uniform deformation effects. The distribution of carbides were affected by localized flow within the strain range investigated at relatively low deformation temperatures 720 - 850°C.

It was found that most of the carbides that precipitate within shear bands resulting from on-going deformation process were Nb- and Mo-rich particles. These are hypothesized to be Nb and Mo carbides. Carbides observed with TEM technique were 20 times smaller to those observed by scanning electron microscope SEM.

A general conclusion follows from the consideration of the role of shear banding in the material structure – this particular mode of deformation may be used as a method of microstructure control. Oriented arrangement of the secondary phases may significantly reinforce the strength of a material without deterioration of the material ductility, as it was observed in case of Fe-Ni [6] and Cu alloys [1-3]. It is interesting to consider a condition when shear bands are formed in deformed material. These localized fields of internal stresses usually show some spatial distribution with long range periodicity. Since shear bands act as preferred sites for nucleation of second phases, the arrangement of the products of decomposition of unstable matrix should also reflect this periodicity. Hence, one may anticipate in the microstructure of the compressed material linear ordering of hardening phases similar to the distribution of shear bands, as well as their significant influence on the mechanical properties of material.

Acknowledgements

This work was carried out with the financial support of the Ministry of Science and Higher Education under grant No. N507 115 31/2788.

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