

# MORPHOLOGY OF $\gamma'$ -PRECIPITATES IN SINGLE CRYSTALS OF NICKEL ALLOYS AT CREEP TEMPERATURES

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## Introduction

The nickel-base alloys are used at high creep-temperatures conditions /1033+1373K/ for such castings like e.g.-blades, rotors and other parts of engines. For these conditions structures and compositions of the alloys should not change during the long time of working.

The metallic materials with similar application divides into two groups: high strengthened superalloys and alloys based on the elements with high melting point /Cr, Ta, Nb, Mo and W/. According to the mechanism of strengthening there are distinguish following types of the alloys [1, 5]:

- solid-solution hardened,
- precipitation hardening,
- strengthening due to second-phase particles.

The discussed alloys belong to the second group of materials and their strengthening effect is achieved by the precipitation of  $\gamma'$  particles. The experimentally determined strength of alloys depends upon such factors as: volume fraction of  $\gamma'$ , radius of  $\gamma'$ , decreasing of ripening rate  $\gamma'$  in higher temperatures than  $0,6T_M$ , formation of  $\delta$ ,  $\eta$  and Laves phases, control carbides and component thickness to grain size ratio [1]. The  $\gamma'$  particles precipita-

te coherently with the austenite-matrix. The composition of austenite includes mostly Ni, Co, Cr, Mo, W, V and  $\gamma'$ -Al, Ti, Nb, Ta so that these particles were identified as  $(\text{Ni, Cr, Mo, Co})_3/\text{Al, Ti, V}$  [2+4]. The nickel-base alloys have thus complex composition and are carefully controlled by at least 10+15 essential elements through melting practice. According to the temperature capability of castings a compositional balance is necessary to achieve all needed properties; that are: maximum creep strength, oxidation resistance, hot-corrosion resistance, microstructural stability in service/because the formation of brittle phases may weaken or embrittle the alloy/ and economical processing. The main purpose in high-temperature applications resolves itself into maximum temperature capability without decreasing other elementary properties.

The strengthening of precipitation hardened nickel-base superalloys is obtained by formation and stabilization in high temperatures coherent particles of  $\gamma'$  [6]. These precipitation increases in flow stress between room and 1400K temperatures and it also possesses inherent ductility. Above  $0,6T_M$  the dependence flow stress-temperature is effected by strain rate, with the change moving to higher temperatures as the strain rate is increased. The strength decreasing is a consequence of disordering about  $\gamma'$  by vacancy diffusion. The suppression of such a diffusion process is impossible to accomplish by changes in  $\gamma'$  morphology or by processing means. The location of the strength drop is not affected by changes of  $\gamma'$ -morphology [8], processing as results on thermomechanical treatments [9] or controlled solidification [10].

The exact temperature at which the peak flow stress is reached is a function of the  $\gamma'$  composition [11]. The temperature dependence of flow stress for single phase  $\gamma'$  alloy increases

with increasing strain rate. According to [12] at a strain rate of 0,03 %/min the peak flow stress is 1071 MPa at 880K but at 3 %/min-1176 MPa at 1019K.

On thermal exposure above  $0,6 T_M$  the  $\gamma'$  particles coarsen at a significant rate. With increasing in size takes place facilitation of dislocation bypassing. For the practical condition minimizing this problem will help retain long-time creep resistance. The coarsening rate of  $\gamma'$  in nickel-base alloys changes with composition. M. Fletwood [7] found that ripening rate of  $\gamma'$  decreased as chromium content increased from 10 to 37%. Similar effect observed [13] at an additions of cobalt, molybdenum and columbium /to about 5% Nb/. The loss of creep resistance during  $\gamma'$  coarsening depends on the volume fraction of  $\gamma'$ . The alloys with low volume percent  $\gamma'$  /M-252/ weakens faster than, for example Inconel 700. Simultaneously flow stress is more sensitive to changes in  $\gamma'$  particles sizes. The main trends in the future of superalloys are increasing in  $\gamma'$  volume fraction and changes in their composition e.g. through slow-diffusing elements such like columbium and tantalum.

#### Experimental procedure

The starting material melted in vacuum induction furnace under the pressure 799,8 Pa. In the same pressure prepared rods, by casting into the metallic forms, with 16 mm o.d. and 100 mm long. This drop-cast master alloy was placed in an alumina crucible and was indirectly heated, using a graphite susceptor in order to minimize induction stirring of the melt. The crucible with the charge were unidirectionally solidified in an induction furnace equipped with a water-cooled bottom chill. Growth rate of the samples were 30,60 or 120 mm/h, which for this set-up was equal to the rate of crucible lowering. After polishing and

etching the samples, an X-ray Laue method was used to determine the growth direction of the crystals/at the surface perpendicular to growth direction/.

Creep and stress rupture properties were determined on specimens of 4 mm diams machined from the single crystals Ni-Cr5-Ti9-Mo6-Al4-B and tested in 1248K at 200 MPa. The mean results are given in Table I. The microstructures examined after unidirectional solidification and creep tests by using optical and also scanning microscop with replica method.

Table I - Rupture life of nickel-

Alloy	Rate of growth mm/h	Rupture life h	Structure
Ni-Cr5-Ti9-Mo6-Al4-B	30	84	bicrystal
	30	84	single crystal
	60	62,5	"
	120	76	"
	120	111	"
Ni-Cr14-W6-Mo3-Ti2-Al2	gravity casting	34	policrystal
		58	
		16	

base alloys after unidirectional solidification.

Results and discussion

The obtained single crystals have an orientation approximated to  $\langle 123 \rangle$  /fig.1/, independent to the rate of solidification.

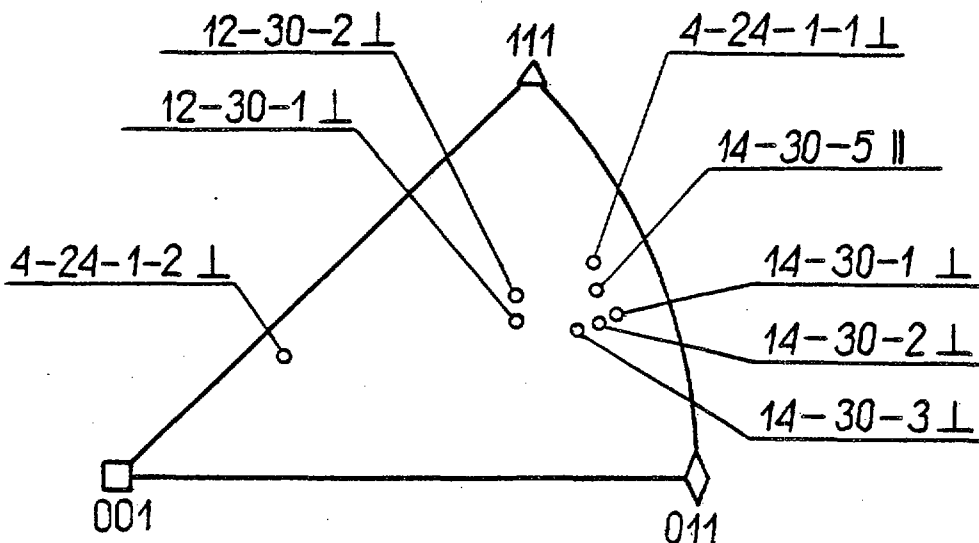
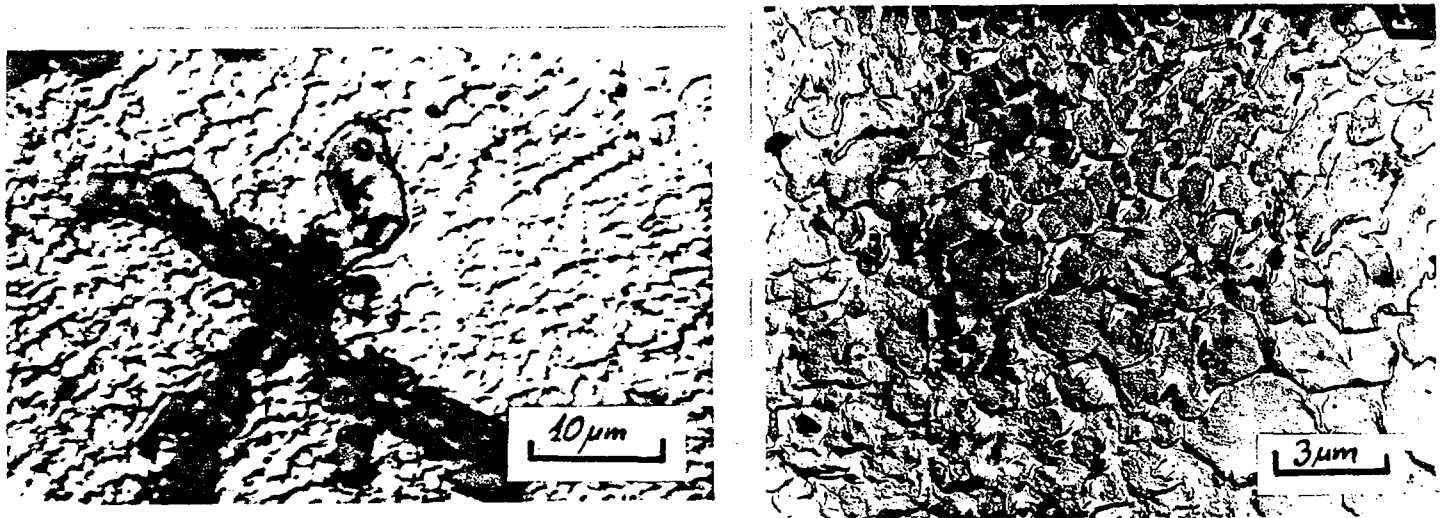


Fig.1. Orientations of examined crystals.

The rupture life of specimens is however markedly differentiated due to the solidification condition. The longest rupture life showed single crystal achieved at growth rate 120 mm/h. Second monocrystal obtained at same growth rate also single crystals grown at 30 mm/h have lower rupture life. In one case obtained columnar structure/two grains/ at 30 mm/h growth rate. Unsteady structure is due to the instability of the gradient temperature at growth time.

The study of the relations between structures parameters and creep properties was made mainly by use of metallographic examination. The data of table I and figures 2+7 show that creep-rupture results depend on such effects like: coarsening of  $\gamma'$  particles in matrix, changes in size and morphology of  $\gamma'$  particles and formation other phases at service conditions /fig.2a/.



a

b

Fig.2 Morphology of  $\gamma'$  particles in tested alloys:  
 a/Ni-Cr14-Ti7-Al4, 1700X  
 b/Ni-Cr5-Ti9-Al4-Mo6-B, 4700X, replica C-Cr

The effect of composition and growth condition on the  $\gamma'$  shape and its distribution is illustrated in fig.2 and 3. In alloy Ni-Cr14-Ti7-Al4  $\gamma'$ -precipitations are uniform arranged with the cubical shape /fig.2a and 3/. In the interdendritic area are located  $\gamma$ - $\gamma'$  eutectic. In the alloy with molybdenum and boron the  $\gamma'$ -particles are less regular /fig.2b and 4/ in the

shape and size. Beside the coarsening of  $\gamma'$  precipitation there are another unprofitable process in the microstructure unidirectionally solidified alloys. This is tendency to formation other phases /fig.2a/. Microprobe studies showed that in their compo-

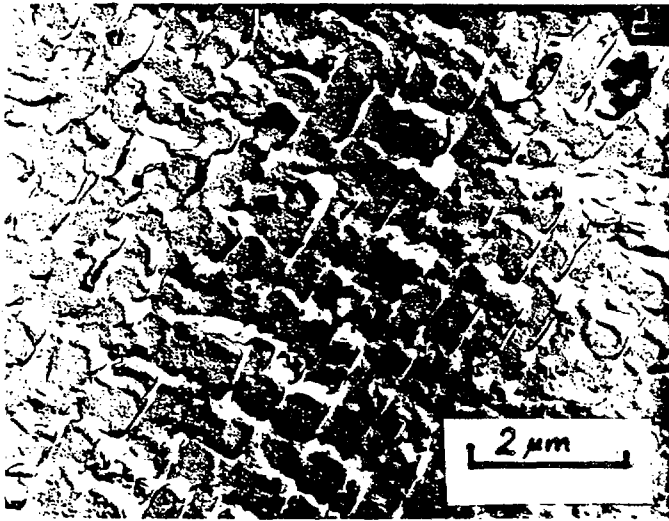


Fig.3. Morphology of particles in the matrix of polycrystal, grown at 120mm/h. Replica C-Cr

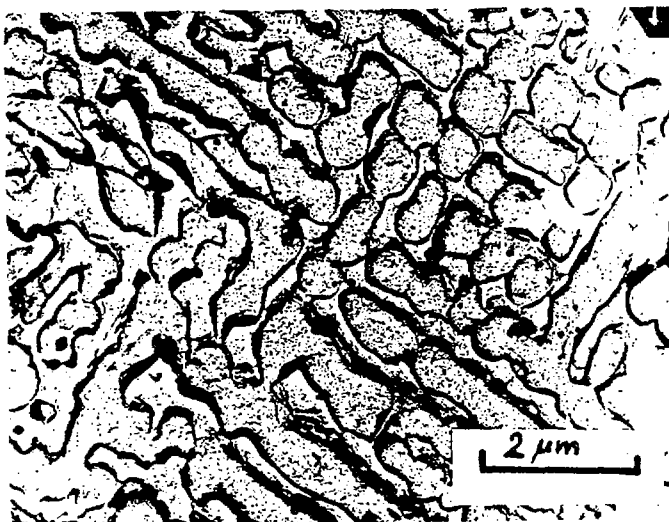


Fig.4. The particles of in the Ni-Cr5-Ti9-Mo6-Al4-B single crystal grown at 30 mm/h.

sition there are 78% of titanium and 6% of carbon. The shape of these compounds, formed in the interdendritic and in matrix places, points out that they are precipitated from the melt before the end of solidification.

The morphology of  $\gamma'$  compared with the microstructure obtained after creep testing showed, that there is relation between rupture life and size of  $\gamma'$  in the matrix. The increase of creep properties is due to the coarsening of  $\gamma'$  phase.

The fine  $\gamma'$  particles gave longer rupture lives /111 hs at approximately

0,5  $\mu\text{m}$  in diameter to 84 hs at 1+2  $\mu\text{m}$  -fig.5-7. The existence of other phases in the alloy has less important meaning. The compari-



Fig.5.  $\gamma'$  and  $\gamma-\gamma'$  eutectic precipitates in sample with 84 hrs rupture life.

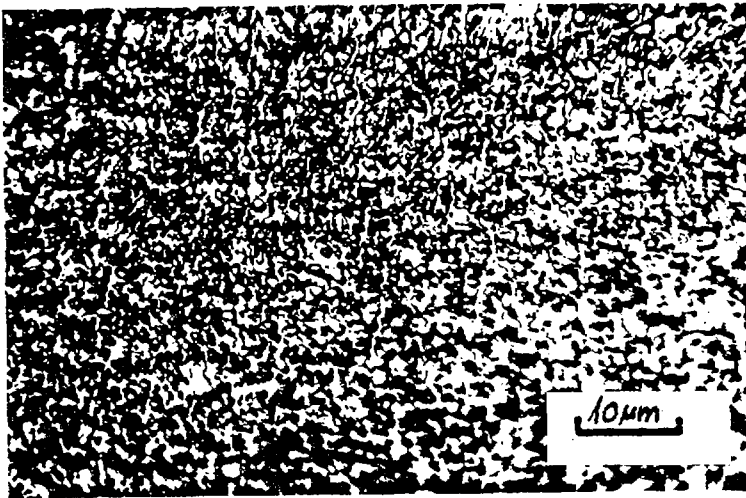


Fig.6.  $\gamma'$ -particles after 111 hrs rupture life

son a creep results between the sample with two grains and single crystals confirms this conclusion /fig.5+6/. The same results are observed between tests in rupture life of single crystals.

#### Conclusion

1. The results of this study show that creep properties are markedly affected by changes in  $\gamma'$  size and their distribution in high temperatures,
2. At the higher growth rates there are greater possibilities to form other phases in the microstructures of the alloys,

crostructures of the alloys,

3. The elimination of grain boundaries by unidirectionally solidification is helpful in creep tests. The rupture life of the alloy Ni-Cr14-W6-Mo3-Ti2-Al2 /used practically/ is two times lower than the same data for nickel-base alloy with molybdenum, prepared in the laboratory.

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