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SOLIDIFICATION AND FRACTURE OF AN AS-CAST Ni ALLOY

STRJEVANJE IN PRELOM LITE NIKLJEVE ZLITINE

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The investigation was carried out on samples cut from 20-kg as-cast ingots of a Ni alloy. The as-cast microstructures were examined, the intensity of the segregations were determined, and the fracture surfaces of the specimens, cooled in liquid nitrogen, were examined. It was found that with slow solidification, carbide particles precipitate on the grain boundaries, diminishing the grain-to-grain cohesion and influencing the morphology of fracture. In the areas of columnar solidification the fracture propagates along the dendrite boundaries.

Key words: Ni alloy, solidification structure, segregation intensity, fracture surface

Izceje v zlitini z lito strukturo poslabšujejo preoblikovalnost v vročem. Za preiskavo so bili odrezani vzorci iz litih ingotov zlitine na osnovi niklja z maso 20 kg. Preiskana je bila strjevalna struktura in določena intenziteta izcej. Vzorci so bili prelomljeni v hladnem in preiskana je bila morfologija površine preloma. Med počasnim strjevanjem nastajajo po mejah zrn karbidni delci, ki zmanjšujejo kohezijo med zrni in vplivajo na morfologijo preloma. Na vrsto preloma vplivajo stebrasta strjevalna zrna zaradi poti preloma vzdolž mej dendritov.

Ključne besede: nikljeva zlitina, strjevalna struktura, intenziteta izcej, površina preloma

1 INTRODUCTION

Most as-cast Ni-based superalloys have a low hot workability[1]. The hot-workability window depends on the microstructural characteristics, the yield strength[2], the deformation rate and the dynamic and static recrystallization. The Ni-based alloy with about 75 % Ni, 20 % Cr, 2.5 % Ti, 1.4 % Al and 0.08 % C is strengthened by sub-micron precipitates of γ' phase (Ni_3Al) and, with a sufficient content of carbon, also by carbide precipitates. During solidification, primary carbide and carbo-nitride particles of the M(C,N) type are formed, while the secondary carbides, M_7C_3 and $M_{23}C_6$, precipitate at dendrite boundaries during the cooling of the solid alloy, because of the decreased solubility of carbon in austenite with decreasing temperature. The presence of carbides and the intermetallic precipitates greatly increases the creep resistance of the alloy, which depends on the size, quantity and distribution of the precipitates. The basic mechanism of hot deformation consists of the gliding and climbing of dislocations. The resulting strain hardening is decreased by dynamic recovery and recrystallization, which also affect the kinetics of precipitation and the temperature of phase transformation. With a higher content of alloying elements, the yield strength of the alloy and the activation energy, E_a, for recrystallization are greater. The yield strength of the alloy also depends on the deformation rate, according to the Zener-Hollomon equation[3] .

For this reason the intercrystalline hollows and segregations decrease the workability until the moment when the microstructure is modified by recrystallization. Sufficient dynamic recrystallization also stops the propagation of the microcracks formed on non-deformable inclusions and on the triple points of the grains. If a grain-boundary crack is oriented orthogonally to the flow of the metal it propagates until a grain boundary of a very different orientation is met, at which point the propagation of the crack is arrested[4].

To obtain the optimal properties for Ni-based superalloys a heat treatment, specific to the particular alloy[5], is necessary. During the heat treatment two essential operations are included:

- The solution of the γ' phase and most of the carbide particles. The solution of the γ' occurs in the range 960–980 °C. This range of temperature increases with the increasing content of Ti in the alloy. $M_{23}C_6$ precipitates are dissolved in the temperature range 1040–1095 °C and M_7C_3 particles in the range 1095–1150 °C.
- The controlled precipitation of γ' and carbide particles during cooling from the solution temperature and during holding at 700 °C.

The two-step heat treatment produces an over saturation of the matrix of the alloy with carbon, which increases the tensile strength at lower temperatures, but leads, however, to a more unstable microstructure at higher temperatures.

The grain size of nickel superalloys also affects the proper relation between the tensile strength and the low-cycle fatigue. The resistance to low-cycle fatigue requires a small grain size (forged material), while the resistance to creep requires a coarse grain size (as-cast material). For this reason, modern turbine blades are mostly produced by casting[6]. As a compromise, a thermo-mechanical treatment giving a "necklace" structure[7] has been developed. This structure consists of coarse, slightly elongated grains, surrounded by small grains, and ensures the best balance of properties.

As-cast Ni alloys usually have a microstructure of coarse, columnar grains, which requires careful starting reductions during the hot-working process. During dendritic solidification, strong segregations of elements are formed in the interdendritic pockets. The intensity of the segregations and the size of the dendrites depend on the rate of solidification: the slower the cooling rate, the coarser are the dendrites and the greater are the segregations.

The aim of this work was to characterise the as-cast microstructure of a Ni-based alloy.

2 EXPERIMENTAL PROCEDURE

The alloy was prepared by melting in air in an induction furnace and casting into 20-kg ingots. The ingots were forged, and during the forging a great number of cracks were formed. Specimens were cut from several places in the ingot and areas containing segregations were assessed by using a scanning electron microscope (SEM) equipped with a wavelength-dispersive spectrometer (WDS). The microstructures were also examined using optical microscopy. The coefficient of segregation was calculated from the difference in the content of elements within the grain and in the interdendritic region. Prior to fracturing the samples were cooled in liquid nitrogen. In addition, the resulting fracture surfaces were examined in the SEM.

3 RESULTS AND DISCUSSION

From the solidification microstructure (**Figure 1**), with coarse, columnar grains near the surface and equiaxed crystal grains in the middle of the ingot, the content of Cr, Co, Al and Ti was determined in the region of the equiaxed grains with point analysis within the grain and in the interdendritic region. The contents of

Table 1: Concentration of elements in and between the solidification grains

Tabela 1: Masni delež elementov v strjevalnih zrnih in med njimi

Element	Concentration, *w*/%		Coef. of segregation $K = c_{max}/c_{min}$
	In the grain	In the inter-dendritic pocket	
Cr	20.08	26.04	1.29
Co	1.14	1.39	1.21
Ni	69.88	69,72	-
Al	0.43	0.46	1,06
Ti	2.03	4.45	2.19
Fe	1.12	1.29	1.15

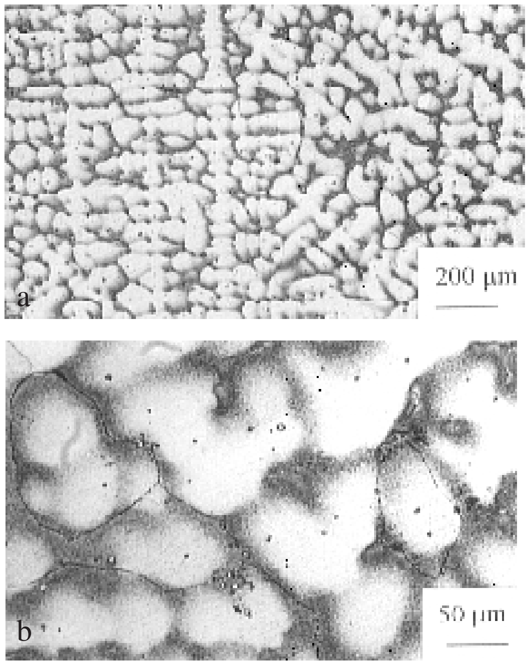

Figure 1: Dendrites and interdendritic segregations of as-cast Ni-based alloy. Etched with Marble's reagent.

Slika 1: Dendriti in meddendritne izceje v liti nikljevi zlitini; jedkano z Marblovim jedkalom

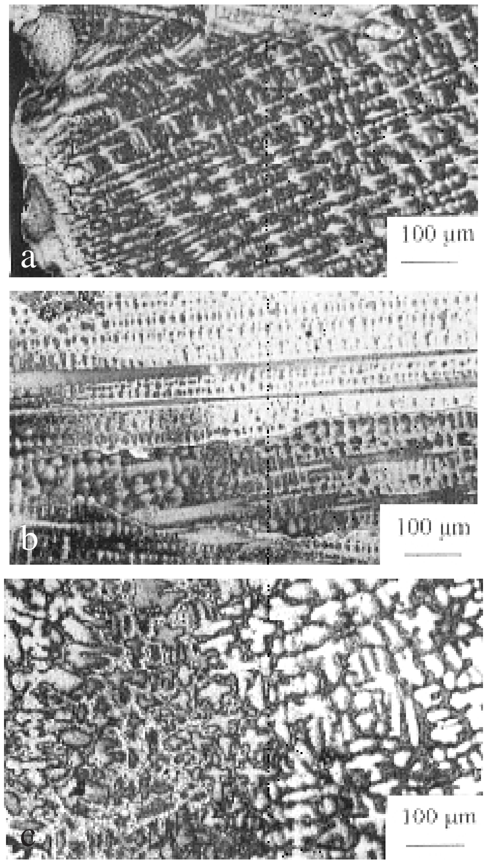

Figure 2: Columnar and equiaxed grains with dendrites in as-cast Ni-based alloy. Etched with Marble's reagent.

Slika 2: Stebrasta in enakoosna zrna z dendriti v liti nikljevi zlitini; jedkano z Marblovim jedkalom

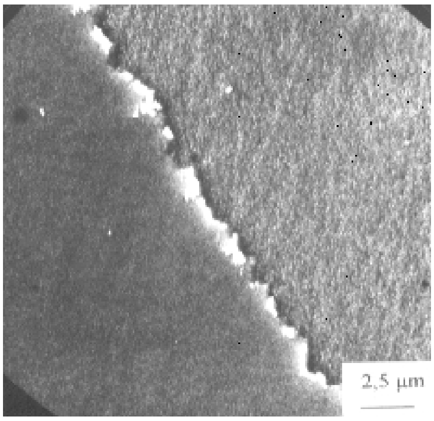

Figure 3: Stringer of carbide particles on the grain boundary in as-cast ingot (SEM)
Slika 3: Niz karbidnih delcev na meji zrn v ingotu z lito strukturo

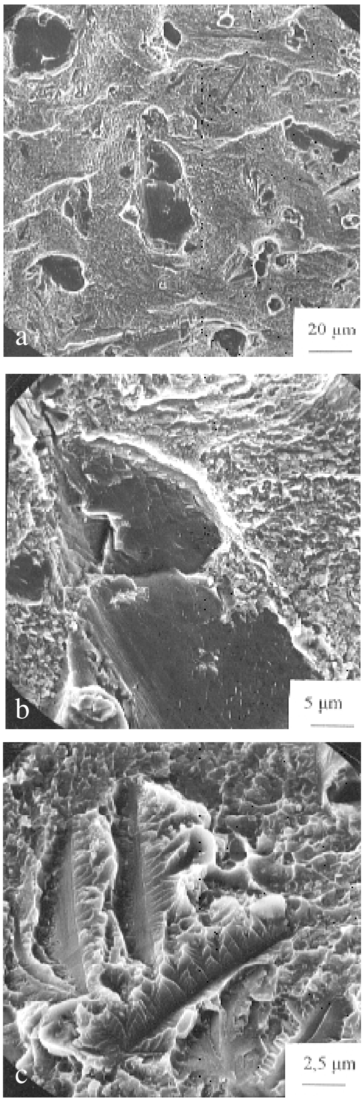

Figure 4: Ductile and brittle fracture of as-cast sample in the region of equiaxed grains (SEM)
Slika 4: Duktilen in krhek prelom v litem vzorcu v območju enakoosnih zrn (SEM)

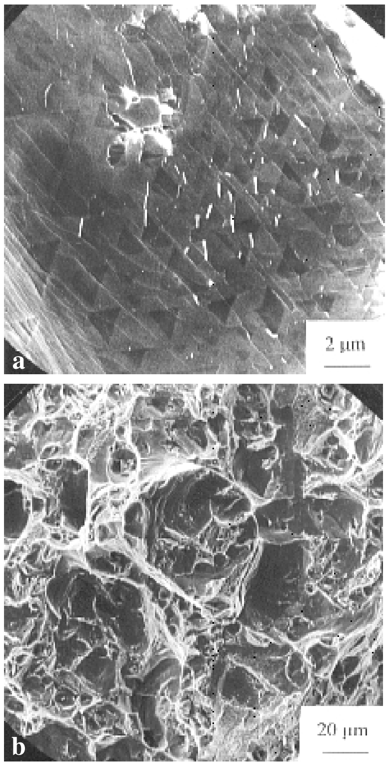

Figure 5: Detail of brittle-fracture surface with triangles and tetrahedra and a mixture of brittle and ductile fracture with dimples (SEM)
Sliki 5: Detajl krhke površine preloma s trikotniki in tetraedri ter duktilni prelom z jamicami (SEM)

analysed elements and the calculated segregation coefficients in **Table 1** confirm the expected strong segregation.

The segregation intensity increases from iron to cobalt to chromium, and it is much greater for titanium. The segregations make the alloy chemically inhomogeneous and cause a difference in the hot deformability.

The morphology of the as-cast microstructure depends on the cooling rate; however, it is also related to the segregation intensity. Several samples were cut from the as-cast ingots and the microstructures were examined

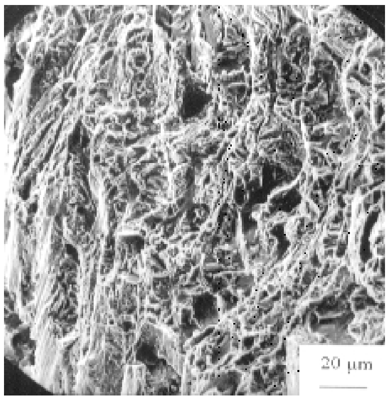

Figure 6: Fracture along the columnar grains
Slika 6: Prelom vzdolž stebrastih zrn

using optical microscopy. The as-cast microstructure reveals a dendritic solidification (**Figure 1**) with coarse, columnar and equiaxed grains (**Figure 2**), with inter-dendritic segregations and stringers of carbide particles on the grain boundaries (**Figure 3**).

The examination of the cold fracture of the as-cast alloy (**Figures 4, 5**) revealed a mixed fracture surface, with both ductile- and brittle-fracture areas being present. Since the alloy is very tough, the fracture was obtained by bending samples that were cooled in liquid nitrogen. As shown in **Figure 5**, on the relatively smooth surface of the brittle fracture, triangles or tetrahedra were observed, indicating that the fracture occurred on the {111} lattice plane. The stacking-fault tetrahedra are special forms of point-defect agglomerates[8]. They are formed by dissociated glide or climb and the aggregation of vacancies. In face-centred cubic metals and alloys the vacancies can group in three-dimensional faults in the form of stacking faults on four {111} planes with six n edges of the tetrahedron.

On the fracture surface a ductile area with trans-crystalline dimples prevails (**Figures 4, 5**), with carbide particles at the bottom of the dimples. The fracture along the boundaries of the columnar grains is similar. The mixture of ductile and brittle fracture is shown in **Figures 4b, c and 5b**. The fractures are mostly trans-crystalline. In some areas a quasi-ductile and quasi-brittle micromorphology was observed, which does not permit a proper interpretation of the prevailing fracturing process.

4 CONCLUSIONS

The Ni alloy had a poor hot workability and a large number of hot cracks appeared during the hot forging. The metallography revealed dendritic solidification with coarse, columnar grains near the surface, equiaxed grains in the centre of the ingot and interdendritic segregation of the alloyed elements and carbide particles on the grain boundaries.

The stringers of the carbide particles are the main reason for the poor grain-to-grain cohesion and represent an easy path for crack propagation during the hot working. They also affect the fracture propagation during cold fracture. The precipitation of carbide particles at the grain boundaries occurs during the slow cooling after solidification or during a too-low soaking temperature prior to hot working.

The examination of the cold fractures revealed quasi-ductile and quasi-brittle areas, partly transcry-stalline and partly intercrystalline. On the brittle-fracture surfaces triangles and tetrahedra were observed, suggesting a fracture on the {111} lattice planes.

For improved hot workability of the alloy it is necessary to decrease the grain size and the amount of segregation and to avoid the precipitation of carbide particles on the grain boundaries.

ACKNOWLEDGEMENT

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