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Recrystallization of Ni-based Superalloy after Cold Deformation

Rekristalizacija Ni-superzlitine po hladni deformaciji

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The strain hardening and isothermal recrystallization after cold deformation of Ni-based superalloy was investigated. Cold deformation below 10% and annealing temperature above 1050°C promote the growth of recrystallized grains. A cold deformation, not lower than 10% and annealing between 1000°C and 1050°C for 30 minutes, produce fine recrystallized grains.
Key words: Ni-based superalloy, cold deformation, strain hardening exponent, statical recrystallization

Izvršena je bila raziskava utrjevanja pri hladni deformaciji in poteka izotermne rekristalizacije po hladni deformaciji Ni-superzlitine. Rezultati kažejo, da končna hladna deformacija pod 10% in temperatura žarenja nad 1050°C pospešujeta nastanek velikih rekristaliziranih zrn. Hladna deformacija nad 10% in 30 minutno rekristalizacijsko žarjenje med 1000°C in 1050°C zagotavljata drobno zrnato rekristalizirano mikrostrukturo.
Ključne besede: Ni-superzlitina, hladna deformacija, eksponent napetostnega utrjevanja, statična rekristalizacija.

1. Introduction

Ni-based superalloys are used in manufacturing of turbine-type machinery, for rotors, vanes and combustion chambers, for exhaust valves in automotive industry, in the tool industry for hot work dies, further in nuclear power plants and for petrochemical equipment as well as in many other places, where a combination of good mechanical properties and corrosion resistance are demanded. Despite of the material development, oriented to Fe-, Ni-, Ti-aluminides and other intermetallics, the Ni-based superalloys remain the base material for use for the critical components (**ref. 1**).

Ni-based superalloys with chromium and other alloying elements are strengthened by precipitation hardening. The matrix is strengthened by precipitation of (Ni$_3$/AlTi/) particles and the grain boundaries by carbide particles, which prevent the grain growth (**ref. 2**).

As cast Ni-based superalloys have a limited hot workability. The hot working is easier in a narrow temperature range above the creep range and below the solidus line, where virtualy no precipitates are found in the microstructures. For most of the alloys with limited hot workability the use of hot extrusion is more suitable especially if it is performed at lower deformation rate (**ref. 3**).

Electric slag remelting also improves the hot workability of the alloy (**ref. 4,5**).

The mastering of the hot working in order to obtain the optimal properties, demands a strict control of the grain size and therefore it is important to control the process of grain growth and the grain size from the solidification to the final cold working. During the hot deformation dynamic recovery and recrystallization occur and cause a much lower rate of strain hardening than is found at room temperature (**ref. 6**). After cold deformation only static recrystallization occurs during the annealing. The aim of the research was to determine the strain hardening at cold deformation as well as to establish the influence of deformation grade and annealing temperature on the start of recrystallization and grain growth.

2. Experimental

The alloy with the following composition: 21% Cr, 1.7% Co, 2.5% Ti, 1.7% Al, 0.62% Mn, 0.72% Si, 0.74% Fe, 0.05% C, bal. Ni, all in wt. %, was melted in induction furnace. The ingots of 60 x 60 mm cross section were cast, electric slag remelted (ESR) into ingot of 100 mm diameter and forged to the bar of 15 mm diameter.

Cylindrical specimens with 13 mm of diameter and length of 10 mm were machined from the forged bar, solution annealed at 1150°C and water quenched. Some samples were continuously compressed with a maximal logaritmic deformation up to 0.9. The exponent of strain hardening (n) was calculated by the method described in **ref. 7**.

Other samples were subjected to 3, 5, 10, 20, 30 and 50% of cold deformation with compression. In both cases a teflon foil was used as lubricant to diminish the friction, between the tooling and the specimen.

After the cold deformation the specimens were isothermal annealed 30 minutes at 900, 1000, 1050, 1100 and 1150°C, water quenched and submited to the examination in optical microscope.

3. Results

The microstructure of solution annealed and water quenched specimen is shown on **Figure 1**.

The strain hardening at cold deformation is shown on **Figure 2**. Factor k_f represents the true yield stress, i.e. the true stress in the sample in the moment of the load action. It was established **(ref. 7)** that the curve of flow stress at compression test in the interval from $\gamma = 0.2$ to $\gamma = 1.0$ can be approximated with the following parabolic function:

$$k_f = k_{f1.0}\,\gamma^{n}$$

The exponent of strain hardening (n) can be estimated from the equation:

$$n = \frac{\ln k_{f0.2} - \ln k_{f1.0}}{\ln 0.2}$$

using the values for $k_{f0.2}$ and $k_{f1.0}$ from **figure 2**. The calculated exponent of strain hardening was n = 0.44. The value for k_f (N/mm^2) can be calculated by using the following equation:

$$k_f = 1770 \times \gamma^{0.44}$$

The logaritmical deformation φ is calculated by the following equation:

$$\gamma = \frac{h_0}{h}$$

where h_0 - represents the initial height of the sample and h - the true height of the loaded sample at each step of deformation.

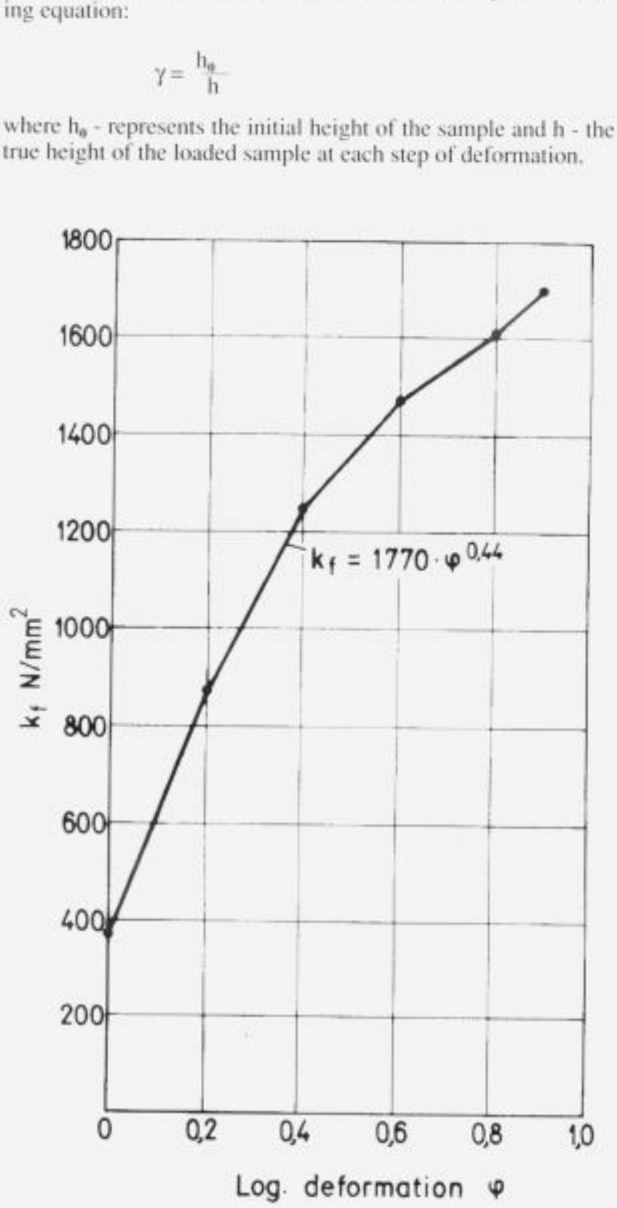

Figure 2: The strain-stress relationship
Slika 2: Odvisnost med deformacijo in silo

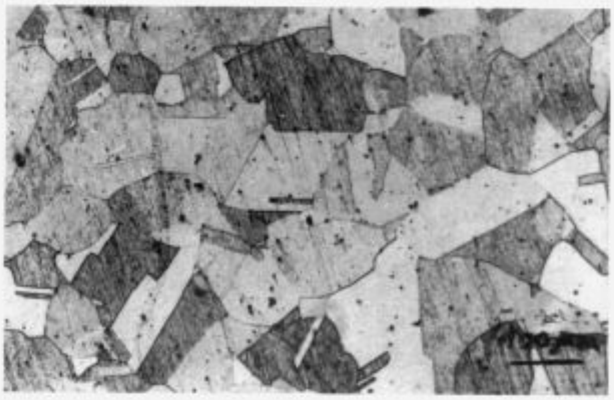

Figure 1: Microstructure after solution annealing
Slika 1: Mikrostruktura po topilnem žarjenju

The microstructure of the alloy after cold deformation and annealing in temperatures range 900 to 1050°C for 30 minutes are shown in **Figure 3, 4** and **5**.

Elongated grains on **Figure 3** show the alloy remain unrecrystallized after annealing at 900°C. At 1000°C the recrystallization occurs by at least 10% of cold deformation.

The lowest temperature at which recrystallization occurs at all grades of deformation is about 1050° C.

At the same annealing temperature the grain size of recrystallized grains decreases with the increasing deformation. At higher temperature the recrystallized grain starts to grow. **Figure 6** shows the connections between the grade of cold deformation and the temperature on the start and advance of recrystallization.

Both, higher annealing temperature and higher cold deformation promotes the start of recrystallization.

The grain size was measured from the micrographs, and represented as ASTM number in **Figure 7**, as relationship between the size of recrystallized grains, the grade of cold deformation and the annealing temperature.

A fine grained recrystallized microstructure can be obtained by at least 10% grade of cold deformation at temperature between 1000 and 1050°C and after 30 minutes of annealing. Higher annealing temperature promotes the grain growth of recrystallized grains.

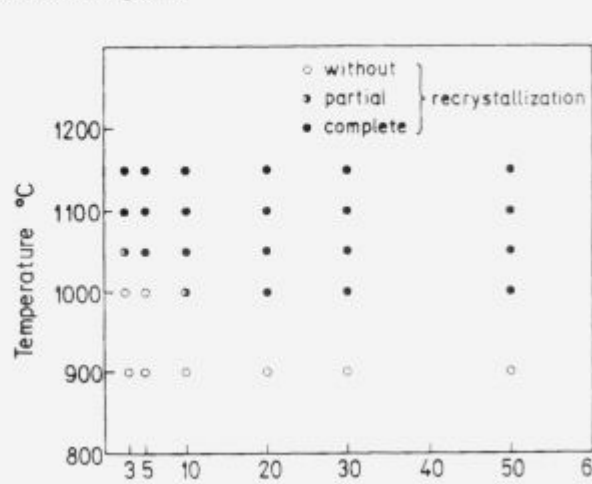

Figure 6: Relationship between the grade of cold deformation, the temperature of annealing and the start of recrystallization.
Slika 6: Povezava med stopnjo hladne deformacije, temperaturo žarjenja in pričetkom rekristalizacije.

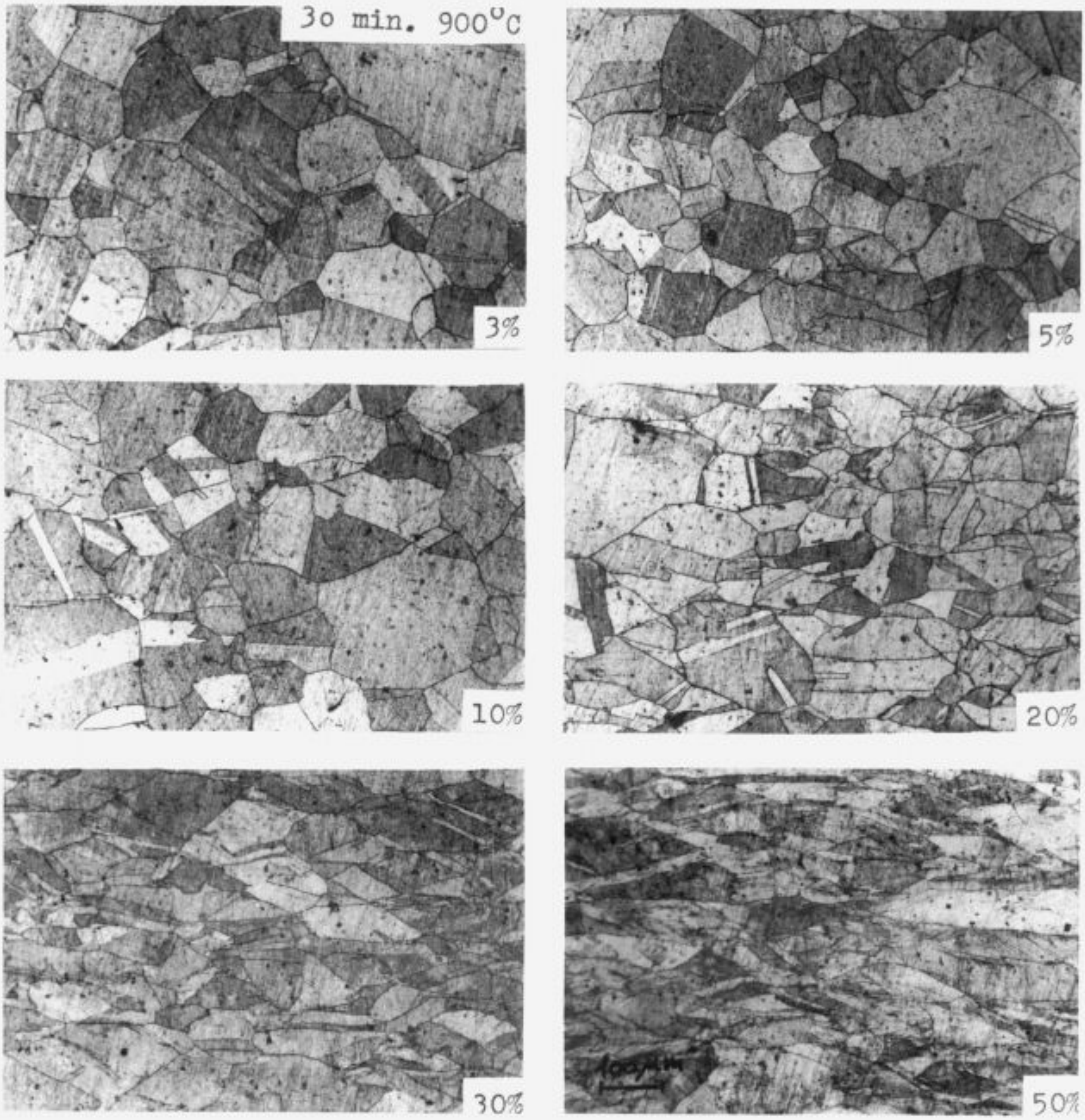

Figure 3: Microstructure of the alloy after cold deformation 3 to 50% and annealing 30 minutes at 900°C.
Slika 3: Mikrostruktura hladno deformirane (stopnja deformacije 3 do 50%) zlitine po 30 minutnem žarjenju na 900°C.

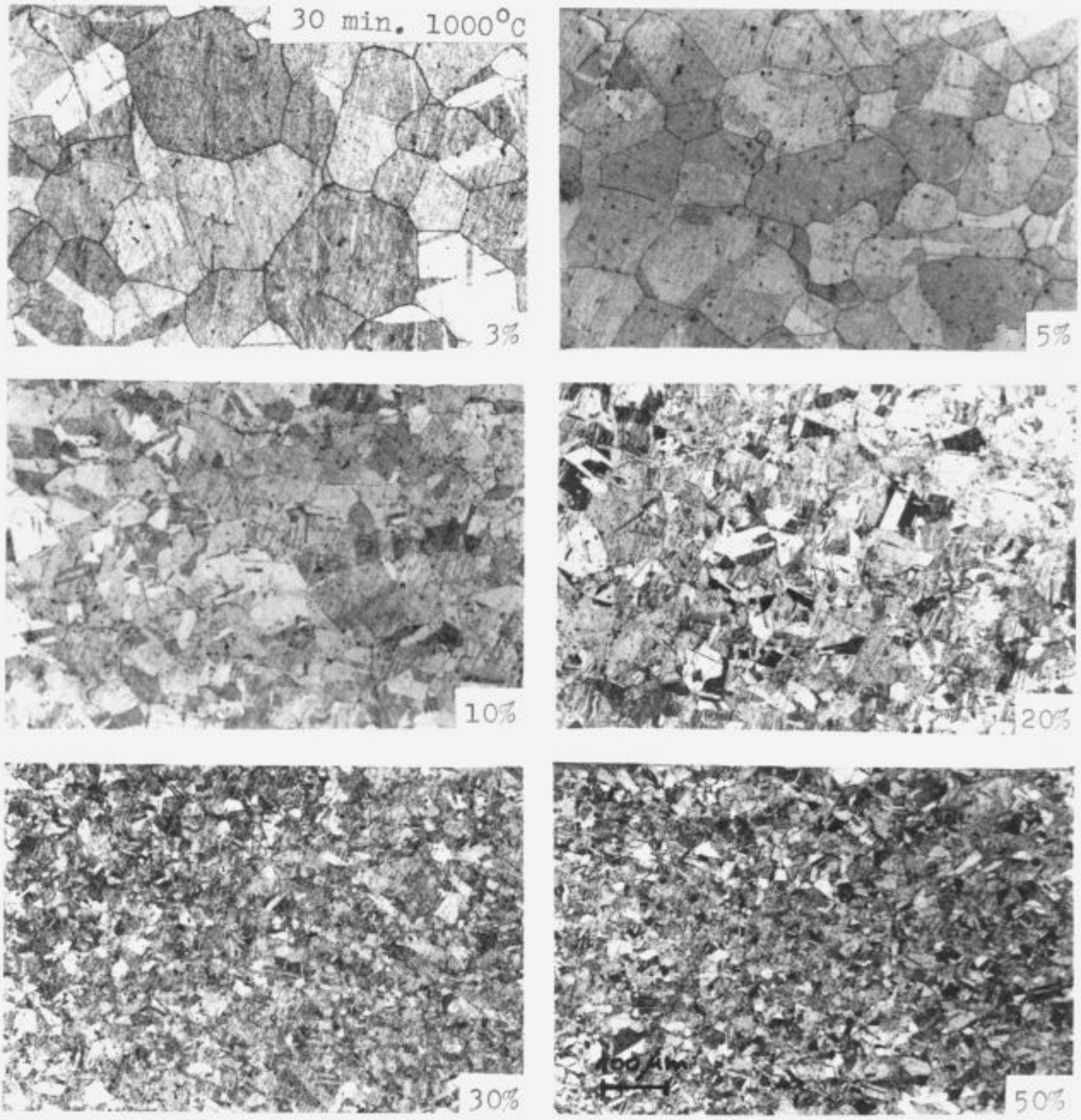

Figure 4: Microstructure of the alloy after cold deformation 3 to 50% and annealing 30 minutes at 1000°C.
Slika 4: Mikrostruktura hladno deformirane (stopnja deformacije 3 do 50%) zlitine po 30 minutnem žarjenju na 1000°C.

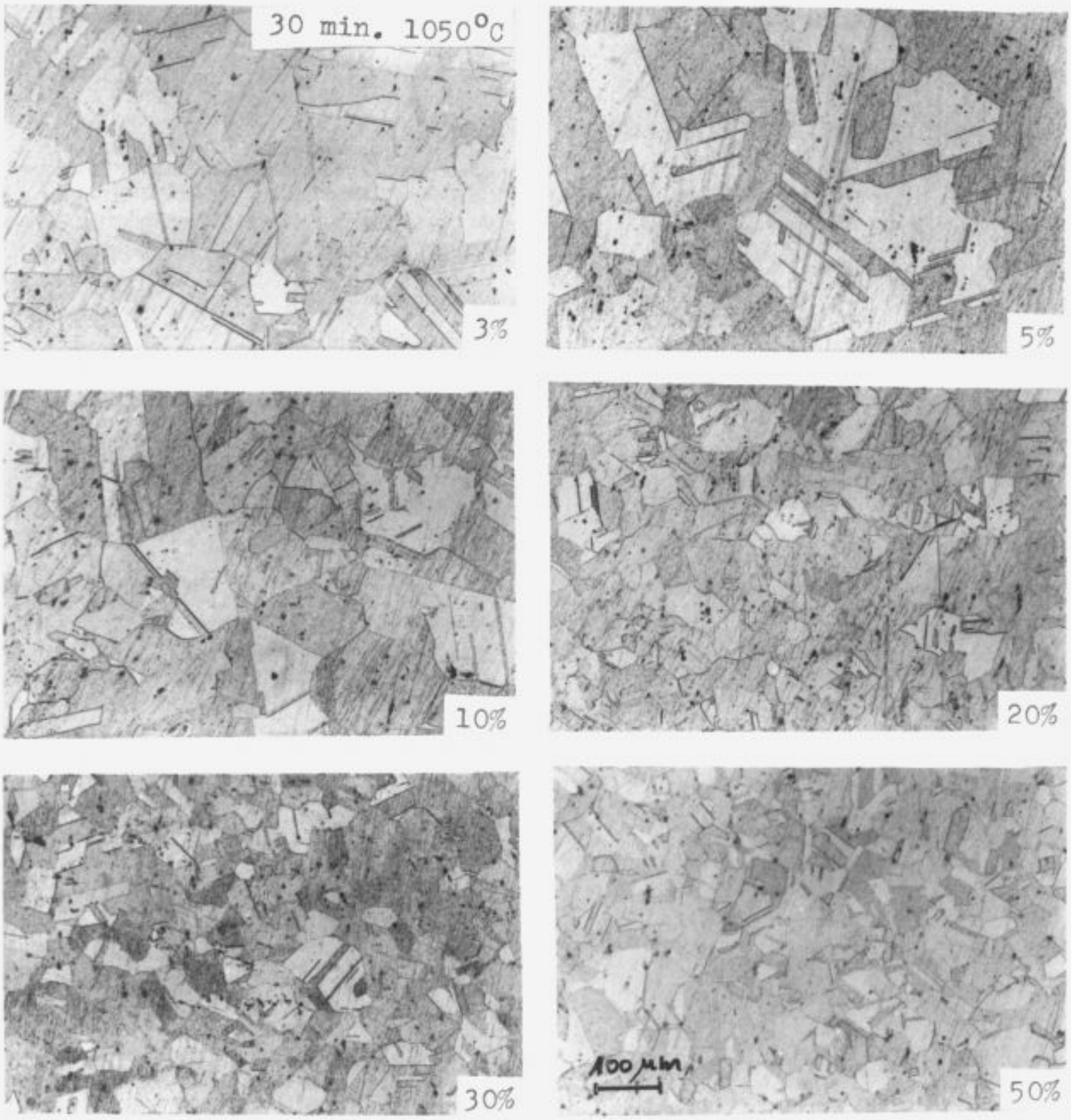

Figure 5: Microstructure of the alloy after cold deformation 3 to 50% and annealing 30 minutes at 1050°C.
Slika 5: Mikrostruktura hladno deformirane (stopnja deformacije 3 do 50%) zlitine po 30 minutnem žarjenju na 1050°C.

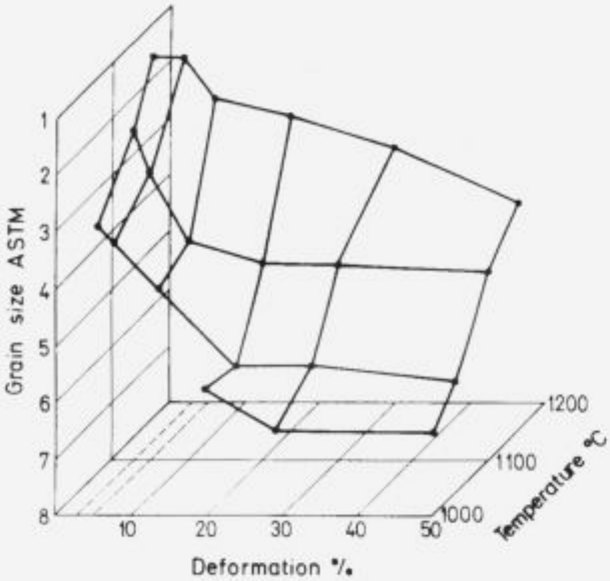

Figure 7: Effect of cold deformation and annealing temperature on recrystallized grain size after 30 minutes of annealing.

Slika 7: Vpliv stopnje hladne deformacije in temperature žarjenja na velikost rekristaliziranih zrn po 30 minutnem žarjenju.

4. Conclusions

Ni-based superalloy was cold deformed by compression test and a hardening exponent n = 0.44 was obtained. The strain hardening can be calculated by the following equation:

$$k_f = 1770 \times \gamma^{0.44}$$

The investigation of the isothermal recrystallization of the superalloy showed that a small grade of deformation (below 10%) and a higher temperature (above 1050°C) of annealing promotes the recrystallized grain growth.

The occurence of partial recrystallization is limited to a relatively narrow temperature range near 1050°C at deformation below 10% and near 1000°C at deformation over 10%.

Finer recrystallized grains of Ni-based superalloy are obtained after a cold deformation not lower than 10% and 30 minutes of annealing between 1000°C and 1050°C.

At annealing above the 1050°C the recrystallized grains start to growth.

5. Acknowledgements

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6. References

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