Microstructure development of Nimonic 80A superalloy during hot deformation

Razvoj mikrostrukture superzlitine Nimonic 80 A med vročo deformacijo

David Bombač[1], Matevž Fazarinc[1], Goran Kugler[1], Savo Spajić[1]

[1]University of Ljubljana, Faculty of Natural Sciences and Engineering, Department of Materials and Metallurgy, Aškerčeva cesta 12, SI-1000 Ljubljana, Slovenia;
E-mail: david.bombac@ntf.uni-lj.si,
matevz.fazarinc@ntf.uni-lj.si, goran.kugler@ntf.uni-lj.si

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Abstract: Uniaxial cylindrical compression tests at various temperatures and strain rates have been performed on the Nimonic 80A superalloy samples in order to define the best hot working characteristics. Evolution of the microstructure in correlation to the deformation temperatures, strain and strain rates has also been investigated by means of optical microscopy. The activation energy for hot deformation was derived with use of the Zener-Hollomon hyperbolic sine equation. Onset of dynamic recrystallization (DRX) was investigated with interrupted compression tests and metallographic analysis. Experimental data was also used for calculation of the processing maps on the basis of Dynamic Material Model.

Izvleček: Pri študiji najprimernejših karakteristik vroče predelave superzlitine Nimonic 80 A so bili izvedeni enoosni valjasti tlačni preizkusi pri različnih temperaturah in hitrostih deformacije. Razvoj mikrostrukture v odvisnosti od temperature deformacije, deformacije in hitrosti deformacije je bila raziskana z optično mikroskopijo. Aktivacijska energija za deformacijo je bila izračunana s pomočjo sinushiperbolične oblike Zener-Hollomonove enačbe. Začetek dinamične rekristalizacije je bil raziskan s prekinjenimi tlačnimi preizkusi in metalografsko analizo. Podatki dobljeni s preizkusi so služili tudi izračunu procesnih map.

Key words: Nimonic 80A, hot working, compression tests, optical microscopy, processing maps

Ključne besede: Nimonic 80A, vroče preoblikovanje, tlačni preizkus, optična mikroskopija, procesne mape

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Scientific paper

INTRODUCTION

Design of new products with nickel-based superalloys and wide field of their use is in constant raise because of very specific material properties. Nickel-based superalloy Nimonic is a group of high temperature alloys intended for sophisticated parts used in high temperature applications. Unfortunately complex system of phases makes them very difficult to deform plastically. Further studies are necessary to predict the best hot workability and final microstructure and therefore a detailed understanding of interactions between hot deformation behavior and softening mechanisms i.e. recrystallization and recovery[1-3].

The Nimonic 80A superalloy is nonmagnetic at room temperature and does not go through any phase transformations when cooled from liquid phase. It contains Ti and Al, that form an ordered g’ phase with composition of $Ni_3(Al,Ti)$ along with MC type primary carbides and Cr rich grain-boundary carbides of $M_{23}C_6$ type. Carbides of MC and $M_{23}C_6$ type were found to precipitate from the matrix at temperatures between 760 °C and 1000 °C, while above 1000 °C main carbides of M_6C type and less stable M_7C_3 will precipitate on grain boundaries where M is usually Cr, and less commonly W, Ta, Nb[4-6, 12].

Best mechanical properties of products made from Nimonic grade superalloy are achieved with homogenous fine grain microstructure. During hot forming metals experience strain hardening as the dislocation density increases. As deformation advances the energy during hot working causes upstart of the softening mechanism like dynamic recovery (DRV) and dynamic recrystallization (DRX). The hot workability of alloy is limited because carbides inhibit DRX and accelerate rupture. To execute the best hot working conditions one needs to select and use correct plastic deformation degree, suitable cool down period and also oversee softening mechanisms.

In this study laboratory compression tests at different temperatures have been performed to define best hot forming characteristics. Evolution of microstructure is investigated with optical microscopy in correlation to temperature, strain and strain rate. Also critical strain for DRX onset is determined from micrographs for various strains. From compression tests processing maps were calculated using dynamic material model.

EXPERIMENTAL PROCEDURE

The polycrystalline wrought nickel-based super-alloy with it’s chemical composition after electro slag remelting presented in Table 1, was supplied by Metal Ravne d.o.o., Ravne, Slovenia. From a forged billet of dimensions 90 mm × 90 mm, which was later rolled to diameter Ø 11 mm and quenched in water from 1030 °C, cylindrical specimens were machined. Size of the

Table 1. Chemical composition of the Nimonic 80A alloy in wt.%
Tabela 1. Kemična sestava zlitine Nimonic 80A v mas.%

	C	Si	Cu	Mn	Cr	Ti	Al	Fe	P	S	N_2 pmm	O_2 pmm
NIMONIC 80A	0.07	0.08	0.02	0.03	19.54	2.38	1.51	0.21	0.002	0.002	70	100

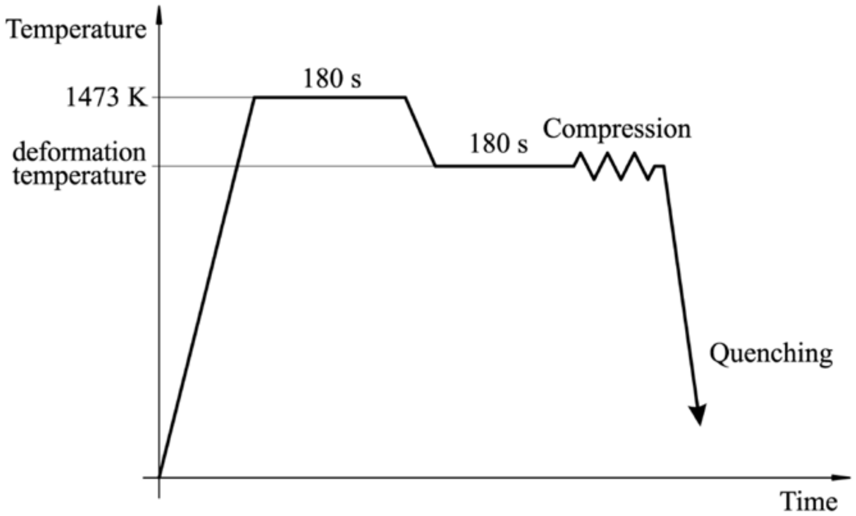

Figure 1. Schematic diagram of the hot compression process
Slika 1. Shematski prikaz procesa toplega tlačnega testa

compression specimens was Ø 10 mm and height 10 mm with measured hardness of approximately 330 HB.

Hot forming parameters and DRX start were studied by means of hot compression tests carried out on Gleeble 1500D thermo-mechanical simulator. Simulation conditions were as follows: temperature 950 °C, 1000 °C, 1040 °C, 1080 °C and 1120 °C and strain rates for all temperatures 0.01 s^{-1}, 0.1 s^{-1}, 1 s^{-1} and 5 s^{-1}. To avoid inhomogeneous deformation, tantalum follies of 0.05 mm thickness were inserted between cylindrical specimen and compression tool–anvil. After deformation the specimens were water quenched to freeze their microstructure. Figure 1 shows schematic time–temperature diagram of the hot compression tests.

Deformed, water quenched samples were visually inspected, cut and prepared for optical microscopy and analysis.

RESULTS AND DISCUSSION

The development of any process modeling capability requires a description of the viscoplastic flow behavior of the material in question. Results of the hot compression tests are depicted in Figure 2. Figure 2a depicts the effect of strain rate on stress-strain curves at 1080 °C. The flow stress decreases with lower strain rates, as does the strain at which the stress peak appears. Typical stress-strain curves for different temperatures and strain rate for strain rate 0.1 s^{-1} are presented in Figure 2b. A clear increase of flow stress with lowered testing temperature can be observed. In general, increase of force is required to deform the specimen at lower temperature and tendency for the phenomenon of interest to occur at higher temperature as strain rate increases.

Initial microstructure of the specimens is shown in Figure 3, where nonuniform grain arrangement with typical anneal twins can

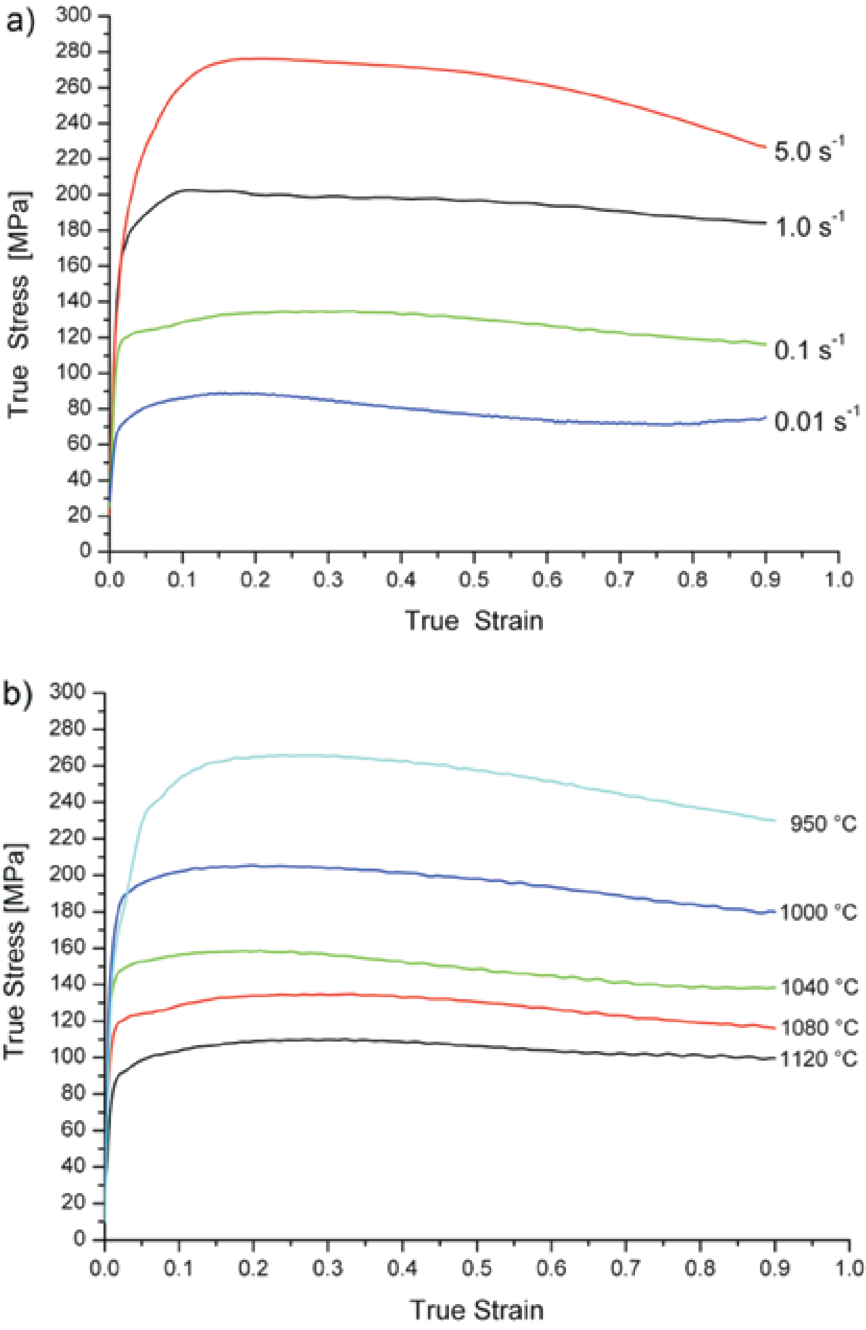

Figure 2. Flow curves; a) at temperature 1080 °C, b) at strain rate 0.1 s^{-1}
Slika 2. Krivulje tečenja; a) pri temperaturi 1080 °C, b) za hitrost deformacije 0,1 s^{-1}

be seen. Grain size varies regarding to the position from where micrograph is collected. On the diagonal and edge of the specimen grains of initial microstructure are finer than in the center.

Values for the peak stress for all deformation conditions were collected and analyzed. The peak stresses exhibits a clear decay trend with higher temperature. As expected, at any given temperature peak stress values also increases with higher strain rate. The activation energy for hot deformation is 379.28 kJ/mol and was derived with the Zener-Hollomon hyperbolic sine equation[7].

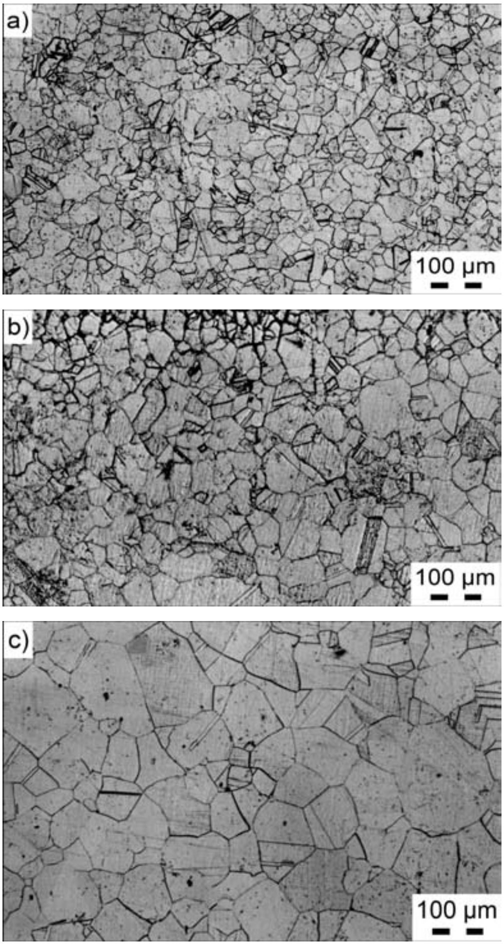

Figure 3. Initial microstructure at different specimen positions; a) diagonal , b) edge, c) center

Slika 3. Začetna mikrostruktura na različnih pozicijah vzorca; a) diagonala, b) rob, c) sredina

Flow curves in Figure 2 exhibits pronounced stress peaks which indicate occurrence of the dynamic recrystallization (DRX), but they does not provide information about the onset of DRX. The critical strain for DRX occurrence can be determined metallographically from grain development at various strains. For this purpose compression test at 1080 °C and strain rate 0.1 s^{-1} was interrupted and microstructure frozen at strain values of 0.0175, 0.175 and 0.40. In Figure 4 micrographs for these strains are depicted. The critical strain for DRX initiation depends on chemical composition, initial grain size, temperature and strain rate. A flow stress increase at diminished rate beyond critical strain, until the work hardening and dynamic softening becomes balanced, climaxing in a peak stress. Grain boundaries are preferential positions for initiation of the DRX[8, 9]. In Figure 4b onset of DRX at grain boundaries with bulging mechanism can be seen. Bulged boundaries later grow into new recrystallized grains as seen in Figure 4c.

Vickers micro-hardness measurements show a difference between recrystallized and non-recrystallized hardness. Micro-hardness of recrystallized grains was between 270 $HV_{0.1}$ and 280 $HV_{0.1}$, while for non-recrystallized grains the value was between 310 $HV_{0.1}$ and 320 $HV_{0.1}$.

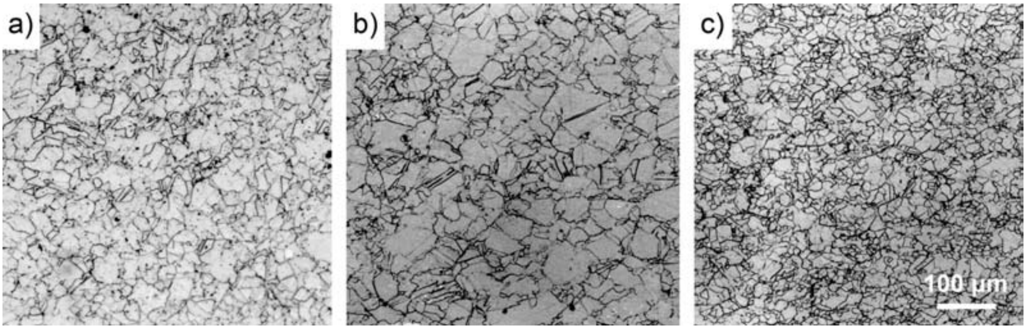

Figure 4. Microstructure observation of DRX onset from quenched samples at temperature of 1080 °C and strain rate of 0.1 s^{-1} at strain; a) 0.0175; b) 0.175 and c) 0.40

Slika 4. Mikrostruktura gašenih vzorcev pri opazovanju začetka dinamične rekristalizacije pri temperaturi 1080 °C in hitrosti deformacije 0,1 s^{-1} za deformacijo; a) 0,0175; b) 0,175 and c) 0,40

Evolution of microstructure obtained with hot compression tests and optical microscopy is shown in Figure 5. Micrographs have been taken from the center part of the specimens. Differences in microstructures can be explained with unequal deformation and stress distribution. At strain rate 0.01 s^{-1}, dynamic recrystallization has been visible at 950 °C, while carbides rearrangement process did not start to this point because of the low temperature and short time cycle. Rearrangement process is clearly visible at other temperatures. At middle temperatures and strain rates, the large un-recrystallized grains are surrounded by small grains, exhibiting a so-called necklace structure. New recrystallized grains nucleate on the boundaries of old ones and grow until energy for grain boundary movement is positive or until they colide with other grains. Start of the DRX with very small amount of recrystallized material volume is visible at highest strain rate. DRX start depends on temperature and strain rate and is shifted to lower temperatures if the strain rate is lowered. Critical deformation for DRX is first obtained at the center of the specimen. These results was also confirmed with findings of other authors[6,10].

Best hot working conditions were established using processing maps calculated on the basis of the Dynamic Material Model[11]. Maps obtained at strains 0.1, 0.2, 0.3 and 0.6 are similar to each other, indicating the limited influence of strain. Processing maps revealed unstable regions at high temperatures and strain rates at strains 0.1 and 0.2 as shown in Figure 6a and Figure 6b, respectively. Nimonic 80A has a prominent high temperature domain with the peak efficiency of power dissipation being about 40 % at 1000 °C and stain rate of 0.01 s^{-1}. For industrial praxis where are higher strain rates, optimal forming interval is between 1000 °C and 1080 °C.

CONCLUSIONS

Retrieved results can be usefully implemented into hot forming process. With

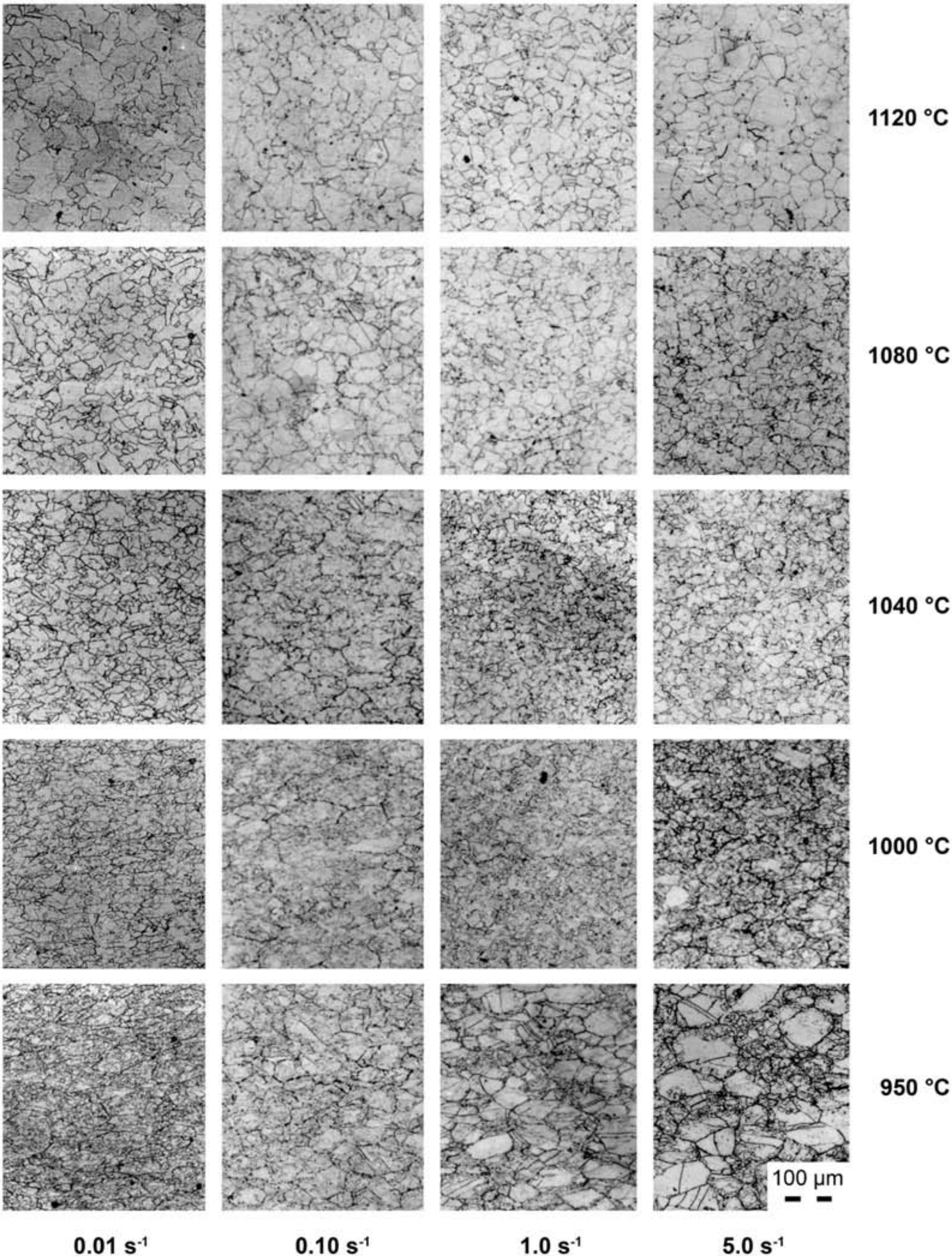

Figure 5. Microstructure development from hot compression tests dependent on the temperature and strain rate, taken from the middle of the specimen

Slika 5. Razvoj mikrostrukture vročega tlačnega preizkusa v odvisnosti od temperature in hitrosti deformacije, s sredine vzorcev

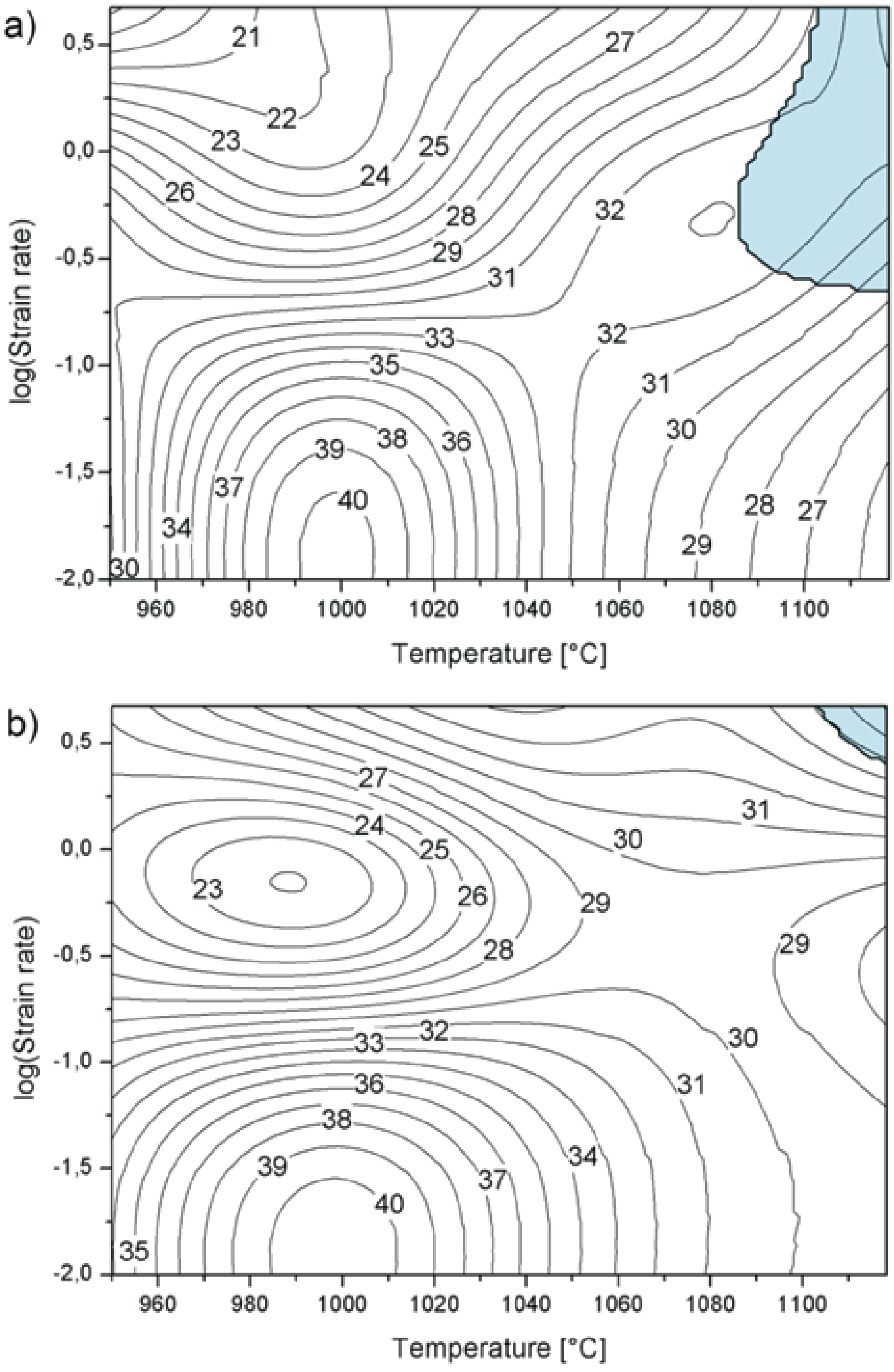

Figure 6. Processing maps for strain; a) of 0.1 and b) of 0.2
Slika 6. Procesna mapa pri deformaciji; a) 0,1 in b) 0,2

correct reductions at the end of the manufacturing process, homogenously recrystallized fine grain microstructure can be assured as has been proved in this paper with hot compression tests at various temperatures and strain rates. The following conclusions can be drawn as a result of this paper.

- When strain rate is increased or temperature lowered, the apex of stress is shifted to higher strain.
- The fraction of recrystallized material is higher at increased temperatures and lowered strain rates. DRX starts at a strain rate 0.01 s^{-1} at 900 °C.
- The optimum hot working conditions for industrial praxis lie between 1000 °C and 1080 °C.
- Unstable regions for hot working conditions were found at strains of 0.1 and 0.2 at high temperatures and high strain rates.

POVZETEK

Razvoj mikrostrukture superzlitine Nimonic 80 A med vročo deformacijo

Pri študiji najprimernejših karakteristik vroče predelave superzlitine Nimonic 80 A so bili izvedeni enoosni valjasti tlačni preizkusi pri različnih temperaturah in hitrostih deformacije. Razvoj mikrostrukture v odvisnosti od temperature deformacije, deformacije in hitrosti deformacije je bila raziskana z optično mikroskopijo. Aktivacijska energija za deformacijo je bila izračunana s pomočjo sinushiperbolične oblike Zener-Hollomonove enačbe. Začetek dinamične rekristalizacije je bil raziskan s prekinjenimi tlačnimi preizkusi in metalografsko analizo. Podatki dobljeni s preizkusi so služili tudi izračunu procesnih map.

Pridobljene rezultate je možno uporabno vključiti v procese vroče predelave. S pravilno izbiro redukcije, še posebej pri koncu proizvodnega procesa lahko zagotovimo homogeno rekristalizirano mikrostrukturo, kot je bilo prikazano s pomočjo tlačnega preizkusa pri različnih temperaturah in hitrostih deformacije v tej študiji. Iz rezultatov študije je možno zaključiti, da kadar je hitrost deformacije povečana ali temperatura znižana, se vrh napetosti premakne proti večji deformaciji. Delež rekristaliziranega materiala je večji pri višjih temperaturah in nižjih hitrostih deformacije. Dinamična rekristalizacija se začne pri hitrosti deformacije 0,01 s^{-1} že pri 900 °C. Optimalni pogoji vroče predelave za industrijsko prakso ležijo med 1000 °C in 1080 °C. Nestabilna področja vroče predelave, je mogoče najti samo pri deformacijah 0,1 in 0,2 pri visokih temperaturah in velikih hitrostih deformacije.

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