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Forged Components and Possibilities for Their Investigation by Neutron Techniques

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A forging process can lead to material's micro- and nano-structure evolution, as well as production of internal residual stresses (RS). Typical fine-grained microstructures are achieved after forging at different temperatures, which are usually investigated and interpreted via conventional tests and analyses including optical, scanning and transmission of electron microscopy. The outputs from these methods can present an important lack of data, regarding basic parameters that aid comprehend and eventually predict degradation, possible fracture and lifetime. These parameters are determinable on real samples and can be supplied by neutron-based non-destructive diagnostic methods, in particular small angle neutron scattering (SANS) for micro- and nano-scale characterisation and neutron diffraction (ND) to assess internal RS.
In this paper, some examples related to forged components - in particular, made of AlSi12CuNiMg alloy - are reported, concerning investigations by neutron techniques. The result can be translated directly into optimization of performances, reliability, design of operating conditions and procedures, in order to achieve a better quality of forged products.

Ključne riječi
Cracks
Forging
Industrial Applications of Neutron
Nanostructure
Residual stresses
Techniques

Otkivci i mogućnosti njihova ispitivanja pomoću neutronske tehnike

Izvornoznanstveni članak

Procesom kovanja može se materijal dovesti do razvoja mikro i nano-strukture materijala, kao i razvoja unutarnjih zaostalih naprezanja. Tipična sitnozrnata mikrostruktura postiže se kovanjem na različitim temperaturama, koje se obično istražuju i objašnjavaju uobičajenim ispitivanjem i analizom uključujući optičke, skenirajuće i transmisijske elektronske metode mikroskopije. Rezultati ispitivanja ovim metodama mogu biti nedostatni za razumijevanje i predviđanje mogućeg propadanja, pojavu loma i skraćenja vijeka trajanja. Ovi parametri mogu se utvrditi na realnim uzorcima nerazornim neutronskim dijagnostičkim metodama, posebno kod malog kuta raspršavanja neutronskog snopa (SANS) za karakterizaciju po mikro i nano-skali te neutronskoj difrakciji (ND), za procjenu unutrašnjeg zaostalog naprezanja..
U ovom radu, neki primjeri se odnose na kovane komponente - osobito od AlSi12CuNiMg legure - iskazuju se, u vezi s ispitivanjem neutronskom tehnikom. Rezultati se mogu direktno optimizirati u smislu poboljšanja svojstava, pouzdanosti, modeliranja radnih uvjeta i procedure, a sve kako bi se opostigla bolja kvaliteta kovanih proizvoda.

Keywords
Industrijska primjena neutronskih tehnika
Kovanje
Nanostruktura
Pukotine
Zaostala naporezanja

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1. Introduction

Mechanical forging allows high quality products to be obtained by means of cold, warm or hot plastic deformations caused by collisions and pressures originating in the forging machines (e.g., open-die, hydraulic radial and mechanical forging presses). The deformations happen freely between flat or very simple shape plates (free forging), or in closed matrixes (fixed forging or pressing). The free forging is carried out on small and large pieces with simple shape, while the pressing concerns relatively small pieces with a complex shape. Temperature is below 3/10ths of the material's recrystallisation temperature (MRT) in cold, above 3/10ths of MRT in warm and above MRT in hot forging. In the pressing process, the material is deformed, until the required shape, between a mould and a dolly that are approached suddenly by a hammer or gradually by means of a press, allowing the production of components with very tight dimensional tolerances. The main feature of the forged pieces consists in their fibrous structure - visible often to the naked eye - corresponding to the orientation of the crystal grains and the fragmented phases. This structure gives the product excellent mechanical properties along certain directions,

Symbols/Oznake

I	- scattering intensity, a.u. - intenzitet raspršavanja	θ	- scattering angle, rad - kut raspršavanja
N	- concentration, cm^{-3} - koncentracija		**Subscripts/Indeksi**
q	- momentum transfer (scattering vector), $Å^{-1}$ - moment prinosa (vektor raspršenja)	G	- gyration - vrtnja
R	- radius, Å - polumjer	T	- total - ukupno
S	- area of interface, m^2 - površina sučelja	inc	- incoherent - nepovezan
V	- volume, cm^3 - volumen	P	- particles - čestice
	Greek letters/Grčka slova	D	- difference - razlika
φ	- volume fraction, % - volumni udio	t	- turbulent - burno
λ	- neutron wavelength, Å - neutronska valna duljina		

with high relation mechanical resistance vs. weight, high tenacity, impact and fatigue resistance. The major fields of applications are automotive and truck, aerospace, agricultural machinery, railroad, valves, defence and energy Industry.

A schematic representation of the main forging processes is reported in Table 1.

Table 1. Main forging processes

Tablica 1. Glavni kovački procesi

<table>
<tr><td rowspan="2">Free deformation without mould / Slobodna deformacija bez kalupa</td><td colspan="2">by flat plates / pomoću ravnih ploha</td></tr>
<tr><td colspan="2">by formed punches / pomoću oblikovanih naprava</td></tr>
<tr><td rowspan="3">Deformation on mould (pressing - relatively small pieces with more complex shape) / Deforamcija u kalupu (tlačenje relativno malih komada složenih oblika)</td><td rowspan="2">by closed matrixes / s zatvorenom matricom</td><td>with formation of burr / s oblikovanjem ruba</td></tr>
<tr><td>without formation of burr (coining) / bez oblikovanja ruba (kovanje)</td></tr>
<tr><td>with open matrixes / s otvorenom matricom</td><td>extrusion / istiskivanje</td></tr>
</table>

The variables associated with the elements characterizing the machining system of mechanical forging are correlated reciprocally and the interactions between the more significant variables are reported in Figure 1.

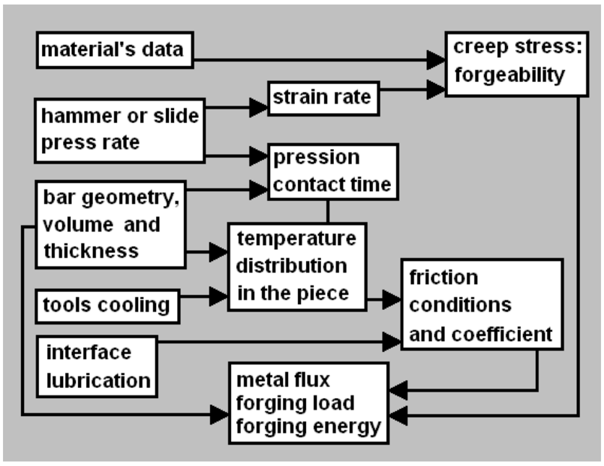

Figure 1. Interactions between the key variables associated with the elements characterizing the machining system of mechanical forging

Slika 1. Interakcije između ključnih varijabli vezanih za elemente koji karakteriziraju obradni sustav obrade kovanjem

Forgings classically exhibit a major grain flow arising from the forced flow of material during mechanical working. The grain results continues throughout the piece, while its strength characteristics are improved. Additionally, the restoration mechanisms together with dynamic recovery and recrystallisation can lead to crystallographic texture and anisotropy of mechanical properties, which consecutively could origin an orientation dependence of RS. In press forging, e.g., as the part cools it becomes stronger and less ductile, with consequent risk of cracking induction if deformation continues [1]. Stresses, strains, micro- and nano-structure play a key rule in the forging process. Machine working rate, moreover, can influence deformation rate and creep stress and, together with the tools` temperature and piece geometry, determines the metal flow, process energy and the forging load.

A list of materials correlated with an increasing degree of forgeability is reported in Table 2. The thermal parameters affecting forging are: temperature, heating procedure, homogeneity of the heating process in the mass of the material under deformation and permanence time, which, associated with the temperature level influences significantly the grain dimensions. Superalloys are hardly forgeable, while cast irons are not forgeable, since they are not deformable. High temperature stability and deformation behaviour of nickel-base superalloys, in particular, depend strongly on microstructural changes caused by complex operating thermo mechanical conditions [2-3]; precipitates behaviour, moreover, depends on temperature and duration of heat treatments [4].

Table 2. Metallic material classification according to forging temperature ranges

Tablica 2. Klasifikacija legura prema opsegu temperatura kovanja

Metal or alloy / Metal ili legura	Machining Temperature range / Temperaturno područje rada, °C
Al alloys	400-500
Mg alloys	250-350
Cu alloys	600-900
C and low alloy steels	850-1150
Martensitic inox steels	1100-1250
Maraging steels	1100-1250
Austenitic inox steels	1100-1250
Ni alloys	1100-1150
PH Semi-austenitic inox steels	1100-1250
Ti alloys	700-950
Fe-base superalloys	1050-1180
Co-base superalloys	1180-1250
Nb alloys	950-1150
Ta alloys	1050-1350
Mo alloys	1150-1350
Ni-base superalloys	1050-1200
W alloys	1200-1300

Carbon steels are sufficiently well forgeable, whereas the stainless steel is forged only in particular cases. In the greater part of moulding processes, the piece temperature is higher than that of the tools, so the metal flow and the mould filling up are influenced by the following parameters: creep stress, forgeability of the piece and complexity of its final geometry, effects of cooling aside of the moulds at the matrix/mould interface and consequent changed wear conditions. Forgeability and grain dimensions increase with temperature; in some materials, temperature decreases by the enhancement of grain dimension; moreover it is influenced by the presence of second phases and the process related stress state. The resistance to deformation increases remarkably if temperature decreases under the minimum, while a risk exists that the material sticks to the mould if the temperature rises above the maximum: in this case, the pieces are rejected and the mould should be restored, with consequent economical damage. Tensile stresses and cracks may occur, in the case of large deformations, corresponding to the part where barrelling happens. To hinder these problems, hydrostatic pressures are usually created to enhance material's forgeability. Austenitizing, followed by hardening and tempering, e.g., is a heat treatment generally carried out on carbon- and alloy-steel forgings. Forged components, nevertheless, can present an RS state and a micro- and nano-structure configuration, leading to prejudices for their performances. A proper investigation of the forged pieces, thus, is essential for their improvement, and a particular advantage can be achieved by using neutron techniques.

2. Methods

The classical investigation methods allow obtaining information, which suffer a fundamental lack of data essential to understand and ultimately forecast ageing, duration and potential failures. These data are well assessable by neutron techniques, in particular SANS for micro- and nano-scale characterisation and ND for internal RS determination. Neutron investigations have lately become an increasingly major probe for materials across a wide range of disciplines and they can reveal significant properties of industrial materials. Neutron techniques, due to their peculiarities, are capable of providing full information down to ~Å (0.1nm) dimensions regarding the micro- and nano-physical structure, investigating from within ~50μm of the surface to a depth of centimetre or still tens of centimetres with a spatial resolution of millimetres. For instance, while X-ray diffraction allows assessing extreme surface RS just to a depth of few hundred Ångström units and only in distinct positions and layer removal methods are unadvisable as they induce additional RS, ND allows determining precisely RS from the surface to depth.

The residual strain assessment by ND is carried out by measurements of angular deviation of the Bragg peak position from its value related to the stress-free state and then, when the diffraction elastic Young modulus and Poisson ratio of the considered material, is known RS can be determined. ND, thanks to its weak interaction with matter, has an elevated test penetration depth and more non-destructive property than other techniques; therefore it is very suitable to measure the global texture of forged components. The theoretical bases of this technique are described in [5-8]. SANS allows a true and full characterization of materials on the nano-scale: averaging over a macroscopic sample volume, it supplies information with high statistical accuracy. The theoretical bases of this technique are described in [5, 8-14].

Models based on the neutron investigation of real samples/components and combined with finite element analysis (FEA), can be developed in terms of dynamic recrystallisation and grain growth phenomena in order to predict material's micro- and nano-structure evolution during forging and also other thermo-mechanical processes. RS, as well as the grain orientation, could be measured by ND, e.g., as a function of time in different regions of the forged component. ND, finally, can facilitate the study of texture evolution during phase transformations in forged materials: the combination of numerical and experimental results is useful to predict the formation of huge crystallographic entities subsequent to forging. The achievable results could aid the quality optimization of forged products, especially in relation to their performances and reliability.

3. Experimental, results and discussions

SANS measurements have been carried out by Rogante Engineering Office at the Budapest Research Reactor, to analyse the microstructure evolution in new and exercised AlSi12CuNiMg alloy components, whose hot die forging was carried out in the temperature range 360-500°C. The working temperature of these parts, in some cases and with relation to certain locations, can go beyond the ageing temperature. Various mechanical characteristics of this alloy, in addition, are highly sensitive to thermal treatments. The same material, among monolithic alloys, exhibits better resistance to thermal cracking than Al25Si and Al-20SiNi alloys [14]. The alteration of mechanical properties due to ageing can be compared with the precipitates size distribution alterations. The characteristic changes of precipitates, in fact, are able to provide practical information, particularly if compared with the results of conventional tests and analyses, for instance micrographs and residual hardness measurements. Micrographs of the considered material have shown an intense growth of precipitates on the grains borders (or regions of dislocations grouping): the arrangement of precipitates, therefore, reflects the grain surfaces geometry (see Figure 2).

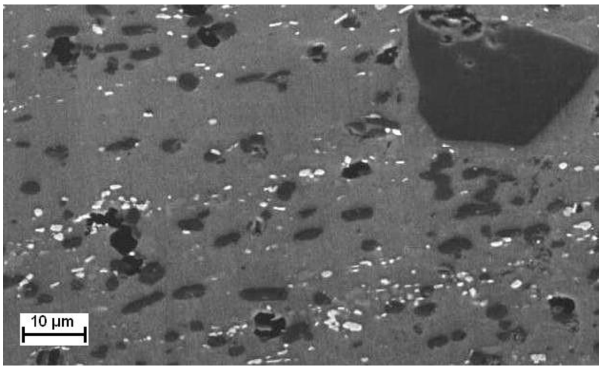

Figure 2. Transmission electron micrograph of AlSi12CuNiMg alloy

Slika 2. Metalografska snimka dobivena TEM mikroskopom AlSi12CuNiMg legure

A coalescence of the hardener elements may happen, in these components, with a mixed hardening mechanism and a precipitation of $CuMgAl_2$, $Mg_2Al_3CuAl_2$ and $CuAl_2$. The last carbide is often a major responsible of the hardening due to precipitation and it is able to improve mechanical proprieties. Comparative SANS analyses of shape and size of these precipitates after different operative periods are helpful to evaluate the ageing process as well as to find the areas of highest thermal alteration. Figure 3 shows SANS patterns obtained for the new and exercised components.

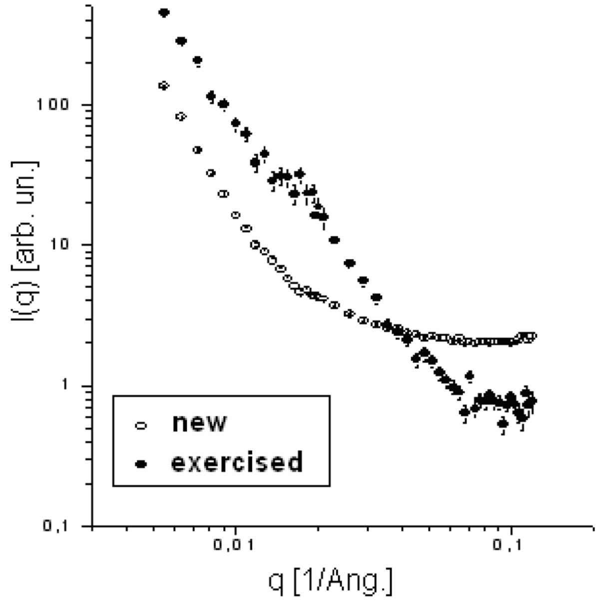

Figure 3. SANS patterns from new and aged AlSi2CuNiMg components

Slika 3. SANS uzorak nove i dozrijevane AlSi2CuNiMg komponente

The data have been achieved in the momentum transfer (scattering vector) range q=0.005-0.12 $Å^{-1}$, where

$$q = (4\pi/\lambda)\sin(\theta/2), \tag{1}$$

corresponding to the spatial scale $R \sim 1/q$ =8-200 Å. An increase of the scattering intensity I at small q~0.005-0.02 Å has been observed for the exercised component, indicating a defect growth on the scale ~100 Å. A weaker scattering has been observed at larger momentum transfer values q~0.1 $Å^{-1}$, indicating the disappearance of small defects such as vacancies annealing, as a result of a thermal effect on the material during the component operation. The search for defects (in particular, precipitates) in the momentum transfer range q=0.005-0.12 $Å^{-1}$ can be considered essential to diagnose early stages of material's ageing. The gain in the scattering intensity at small angles, in the considered case, is clearly visible in the behaviour of the ratio $R(q) = I_{EXERCISED}/I_{NEW}$ (Figure 4).

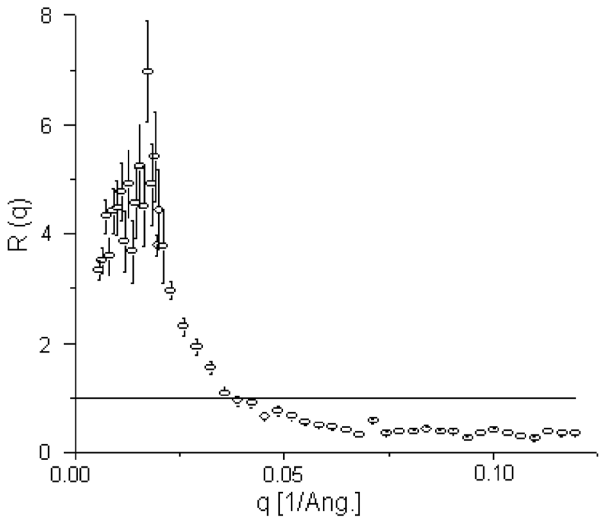

Figure 4. Behaviour of the ratio $R(q) = I_{EXERCISED}/I_{NEW}$

Slika 4. Svojstva omjera $R(q) = I_{EXERCISED}/I_{NEW}$

The intensity for the exercised component is enhanced by a factor 3 to 4 ($q \sim 0.01$ $Å^{-1}$). Some substantial structural changes have been observed even at small scales ~10Å, apart from an intense growth of nano-scale defects (≥100Å, Figures 3 and 4). Another representation, the Porod plot (Figure 5) helps the understanding in detail of these material structure transformations, showing a crucial difference between the structures of new and exercised components.

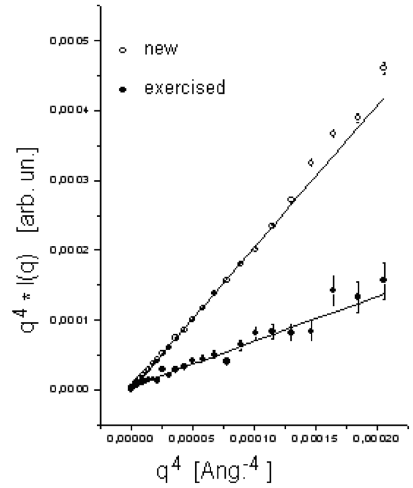

Figure 5. Porod approximation

Slika 5. Porod approksimacija

The intensity data $I \cdot q^4$ for both new and used materials obey the linear function $I \cdot q^4 = A + Bq^4$ in the range of q^4 =(2-12)10-5 $Å^{-4}$. The fitting parameters for the Porod approximation are listed in Table 3.

Table 3. Porod approximation parameters for AlSi2CuNiMg components

Tablica 3. Parametri Porod aproksimacije za AlSi2CuNiMg komponente

component / komponenta	$A \cdot 10^6$, arb. un.	B, arb. un.
new / novo	0.64 ±0.60	2.04 ±0.02
exercised / primjenjeno	4.44 ±1.16	0.65 ±0.04

The first parameter $A \propto 2\pi(\Delta K)^2 \cdot S_T$ is proportional to the total area of particles (e.g. for spheres $S_T = 4\pi R_P^2 N_P$, where R_P is the radius and N_P is the number of particles in the sample). The second parameter $B \propto d\sigma_{inc}/d\Omega \approx B_{Al}^2 \cdot N_{Al}$ is the measure of incoherent scattering mostly reflecting nuclei concentrations and scattering lengths. A strong increase in defects' surface (factor ~7) and a simultaneous decrease of incoherent background indicate an intense formation of internal surface via integration of small defects (disappeared). The differential signal $I_{EXERCISED} - I_{NEW}$ has been obtained using the parameters of Table 3 and having subtracted the incoherent background (see Figure 6).

The pattern in Figure 6 shows an intense growth of defects as thermal treatment result due to the exercise of the investigated component. The difference $I_D = I_{EXERCISED} - I_{NEW}$ really represents the nano-scale precipitates grown in the material. This differential intensity demonstrates primarily the contribution of the defects ~100 Å. The same plot shows the particles as themselves and their clusters as revealed at small angles. The intensity distribution is reasonably described by a three component Guinier function (see Table 4).

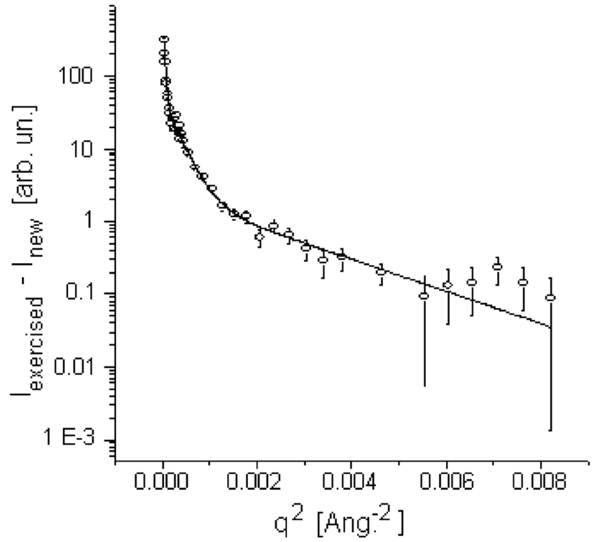

Figure 6. Guinier approximation

Slika 6. Guinier-ova aproksimacija

Table 4. Parameters of three component Guinier approximation

Tablica 4. Parametri trokomponentne Guinier-ove aproksimacije

I_{o1}, arb.un	R_{G1}, Å	I_{o2}, arb.un	R_{G2}, Å	I_{o3}, arb.un	R_{G3}, Å
2.3 ±0.5	39.0 ±3.0	49.1 ±5.1	104.5 ±4.0	829 ±149	343 ±18

In the diagram of Figure 7, finally, the magnitudes $I_{oi}/R_{Gi}^3 \propto \varphi=N_i{\cdot}V_i$ are plotted, which are proportional to the component's volume fractions.

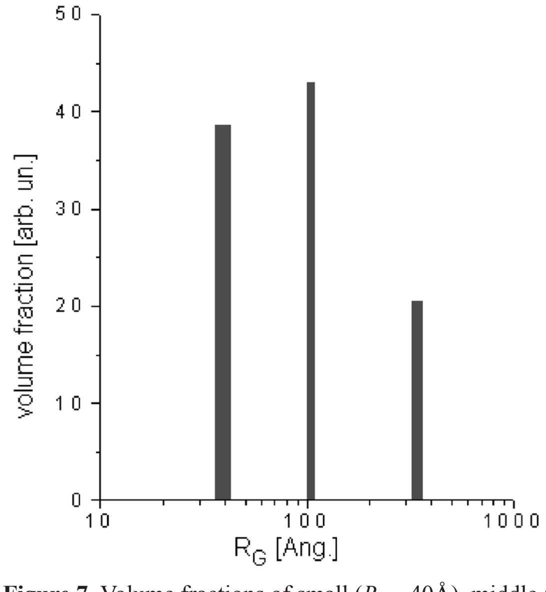

Figure 7. Volume fractions of small (R_G ~ 40Å), middle (R_G ~100 Å) and large defects (R_G ~200-400Å) grown.

Slika 7. Volumenski udio malih (R_G ~ 40Å), srednjih (R_G ~100 Å) i velikih grešaka (R_G ~200-400Å)

Various other examples related to neutron investigation of forged components are reported in [4, 15]. A NiCrMoV forged wheel of an axial compressor for a heavy duty gas turbine was considered for RS determination by ND and for a SANS nanostructure investigation in correspondence of specific positions individualized from a previous FEA, to perform comparative studies of the expected changes of the precipitate distribution with the results of the RS investigation. ND, in particular, allowed measuring radial and hoop RS (see Figure 8), revealing that the fatigue of the wheel is also a lifetime limiting factor [16]. SANS investigation, on the other hand, showed that the material possesses an isotropic nanostructure composed of tiny domains (precipitates, diameter ~ 200-300 Å), concentration N≈(1-3)·10^{14} cm^{-3}, volume fraction φ ≈0.1-0.2 % and total area of interface S_T ≈ 0.2-0.4 m^2. The SANS-data testified a low concentration of nano-defects in the wheel's material, as compared to steels after thermal treatment, in which an intense formation of precipitates with volume fraction exceeding ~ 1 % is usually induced [17]. Various other studies and feasibility applications of ND and SANS related to forged materials and components can be mentioned as follows. Deformed metallic alloys were analysed by SANS (the size of defects being ~100 nm), especially in the fracture zones. The results showed that the main sources of scattering are micro-cracks and precipitates and that plastic deformation of metals for a few tens percent gives rise to huge scattering intensities several times higher than those existing in the original samples (Cu, Al and Ni) [18]. A quenched IN718 aero-engine compressor disc (weight ~40 kg, diameter ~400 mm and maximum thickness ~45mm) produced by forging was investigated by ND in order to measure RS, whose hoop and radial components resulted in compression at the surface (up to -600 MPa) and tensile at depth (up to 400 MPa) [19]. RS were analysed by ND in a number of identical hot-forged water quenched IN718 compressor discs and the results agree with those obtained from FEA [20]. The through-thickness RS distributions were determined by ND within three 120mm thick 7449 aluminium alloy rectilinear forgings with similar geometry. Results indicated large magnitude (>250 MPa) tensile RS in the centre of an as quenched forging, balanced by surface regions stressed in compression (<-200 MPa). Cold water quenching these forgings resulted in biaxial surface compressive stresses in the range 250-300 MPa, balanced by inner tensile stresses up to 350 MPa [21]. Through-thickness RS measurements by ND were carried out in two 215 mm thick heat treated rectilinear Al7075 and Al7010 alloy forgings. 7075 is a much more quench sensitive alloy when compared to 7010, whose forging exhibited considerably larger tensile stresses in the core. The RS distribution was found to be comparable for both alloys changing from highly tri-axial and tensile in the core to biaxial compression in correspondence of the surface [22]. Few works have been performed on studying the texture evolution of forged particulate reinforced magnesium matrix composites by ND. The as-cast ingots of SiCp/AZ91 magnesium matrix composite produced by stir casting were cut into cylindrical billets and then forged at different temperatures (320, 370, 420, 470 and 520 ◦C) at a constant RAM speed of 15mm/s, with a 50% reduction in height. The texture of these forged composites was measured by ND. The results showed a characteristic forging texture with the normal direction of the basal plane turning to forging direction clearly during the whole forging process.

dissimilar from the transformation processes in the Ni-Mn-Ga alloys with higher martensitic transformation temperatures [24]. ND was adopted to investigate the bulk textures of HSF-Bi2223 superconductors' samples formed by hot stacking-forging process. The texture data showed extreme intensity variations as the sample was tilted and rotated azimuthally in relation to the neutron beam. The obtained information helped to explain anisotropy, superconducting properties and microstructure observations related to the investigated samples [25].

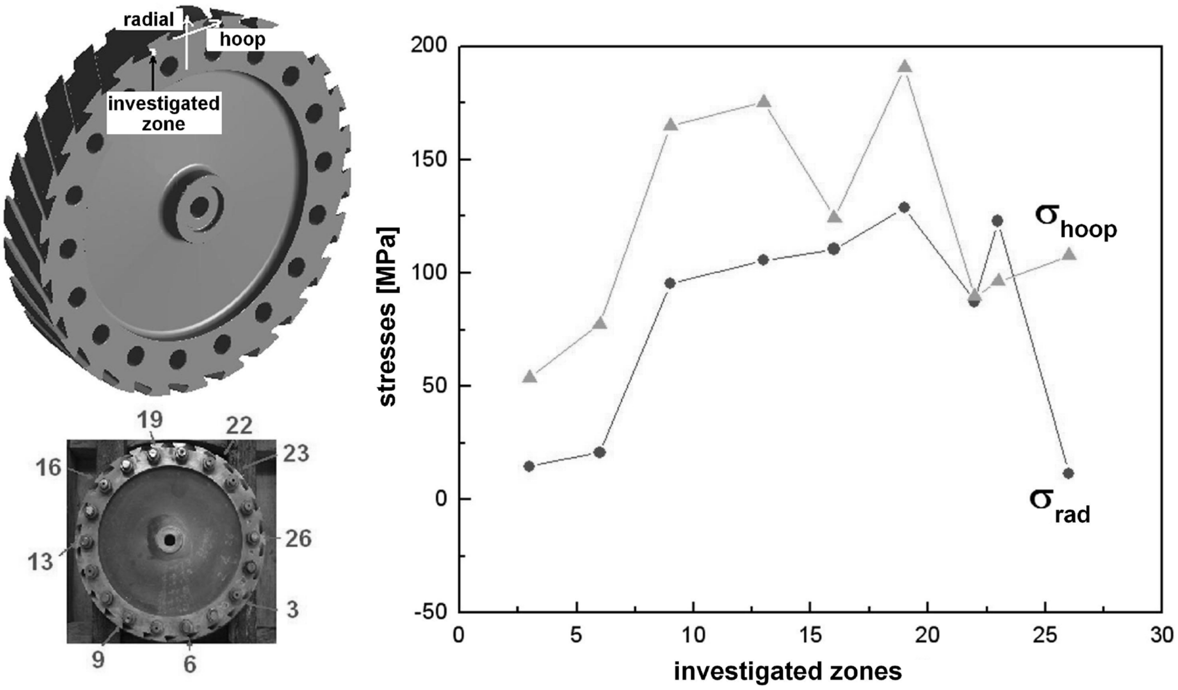

Figure 8. Residual stresses measured by neutron diffraction in a NiCrMoV forged wheel of an axial compressor for a heavy duty gas turbine [15]

Slika 8. Zaostalo naprezanje izmjereno pomoću neutronske difrakcije na kovanom rotoru aksijalnog kompresora plinske turbine za teške uvjete rada iz legure NiCrMoV [15]

A strong basal plane texture was found in the matrix of composite with the basal plane perpendicular to forging direction; the intensity of the basal plane texture appeared weakened by increasing the forging temperature [23]. The crystal structures - both in austenitic and martensitic states - of a hot forged ferromagnetic shape memory alloy (SMA) of $Ni_{48}Mn_{30}Ga_{22}$ prepared by induction melting, were investigated by ND, in order to study the strong textures the large anisotropy in plane plastic flow developed during the forging process. The results supplied also information on structure changes by cooling the considered material up to 243 K and successively to 19 K, indicating, in particular, that no inter-martensitic transformation exists in the investigated alloy, which is

A co-deformed Cu-10vol.% Cr composite produced from cast Cu-Cr by hot forging and then cold swaging to a reduction of 95.6% (to achieve a fibrous reinforcing microstructure) was tested by ND in order to measure the changes in elastic strain with applied tensile loading in the axial and transverse directions. The results showed that the Cr phase deforms more than the Cu phase, reaching the yield locus at approximately the same strain as the Cu phase, due to the co-deformation previous to testing and to a mixture of its elevated stiffness and the possible existence of swaging induced RS [26]. An unalloyed Al matrix reinforced by spherical Al–Cu–Fe alloy particles consolidated by vacuum hot pressing and quasi-isostatic forging was tested by ND to determine RS. Composites reinforced by 15, 20, and 30 vol.% particles were investigated and the results showed that those consolidated from Al and $Al_{63}Cu_{25}Fe_{12}$ quasi-crystal alloy powders possess compressive RS in the Al matrix, probably due to the volume expansion of the reinforcement particles during the phase transformation and to the stiffness mismatch of the matrix and reinforcement phases [27].

4. Conclusion

Neutron techniques, as non-destructive diagnostics, can be helpful to investigate forged components, supplying significant data on basic parameters connected with degradation, fracture and other phenomena and allowing more reliable lifetime assessments.

Key works have been individuated and reported in this paper, showing a sort of state-of-the-art and also demonstrating the complementarity between neutron-based methods and the other techniques traditionally used. In particular, defect evolution in new and exercised AlSi12CuNiMg forged components were analysed by SANS. Precipitates' intensity distribution and volume fractions were determined and a significant growth of the defects ~100 Å together with the loss of small defects such as vacancies annealing was detected, due either to the exercise or to thermal effects; important differences between the structures of new and exercised components were also found.

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