

TERMIČKI INDUKOVANE STRUKTURNE TRANSFORMACIJE AMORFNIH LEGURA NA BAZI GVOŽĐA

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Cilj predavanja

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Ispitivanje termičke stabilnosti

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Uticaj termalnog tretmana na mikrostrukturna svojstva legure

Kinetika termalne stabilizacije

Korelacija kinetičkih parametara sa mikrostrukturnim parametrima



Difraktometar X-zraka



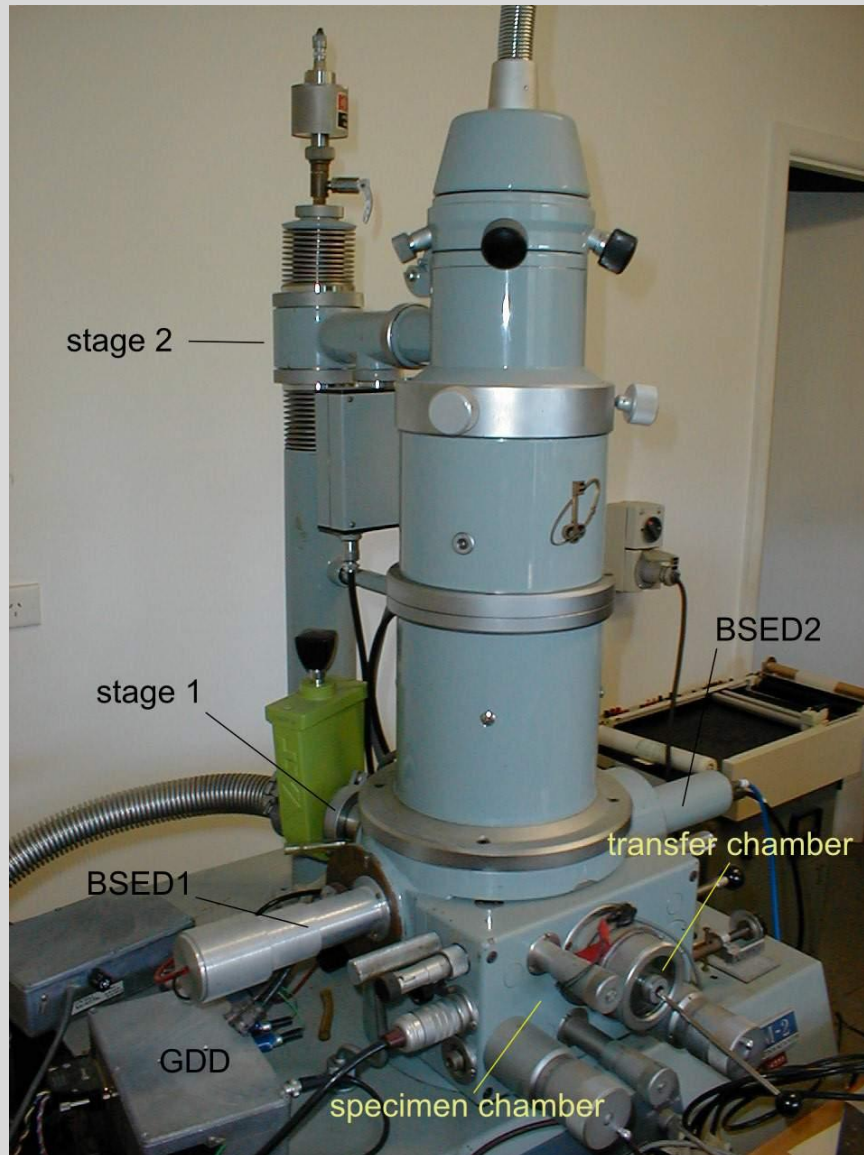
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SEM

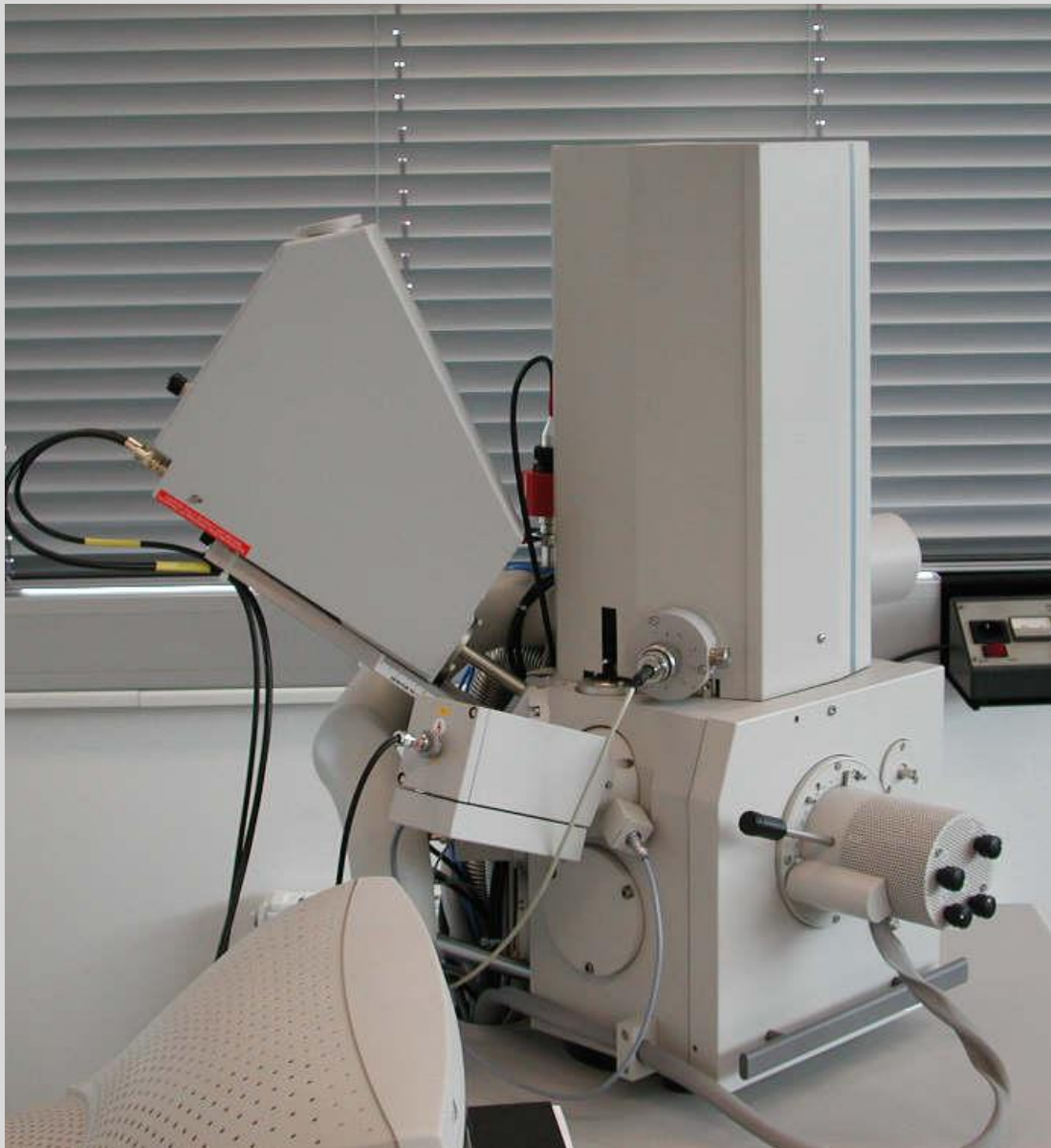


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SEM



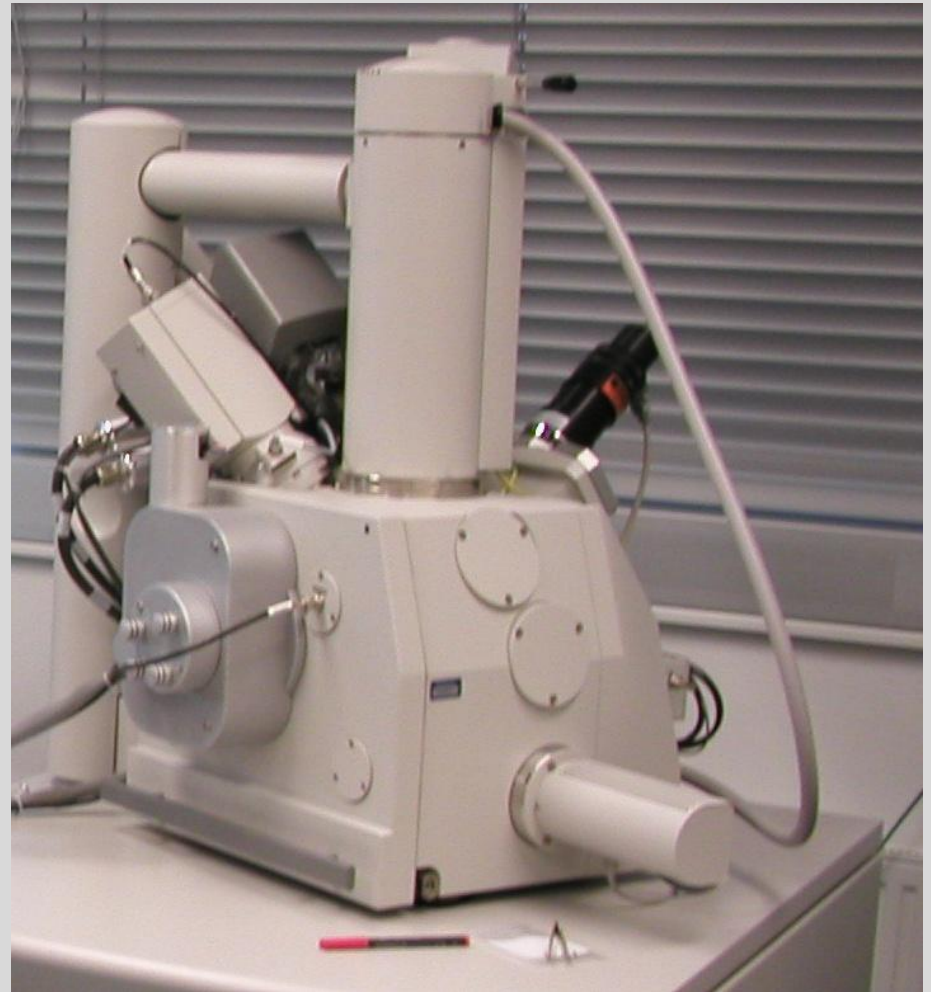
ESEM



ESEM



FIB



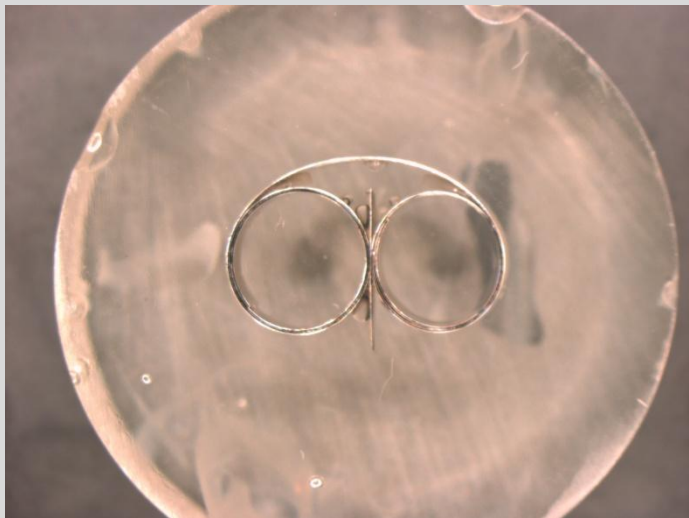
FIB



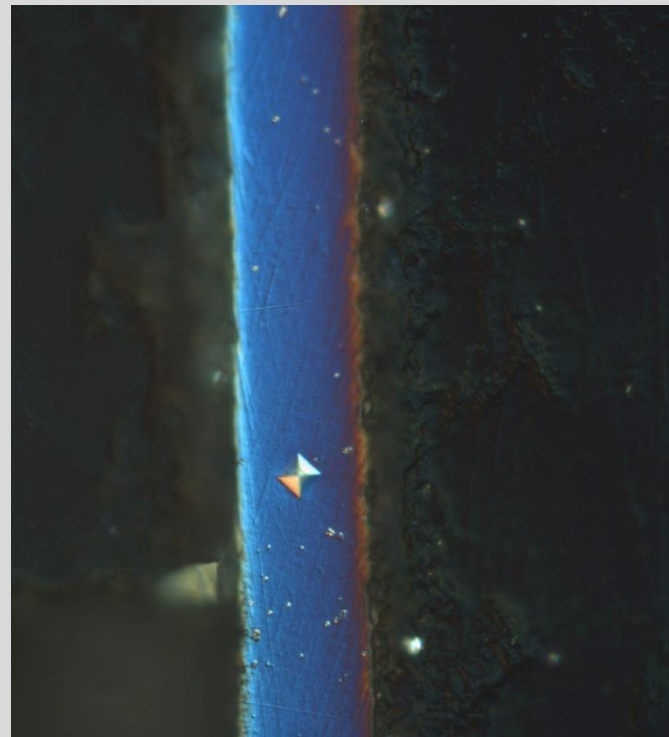
Vickers taster



Dualindent

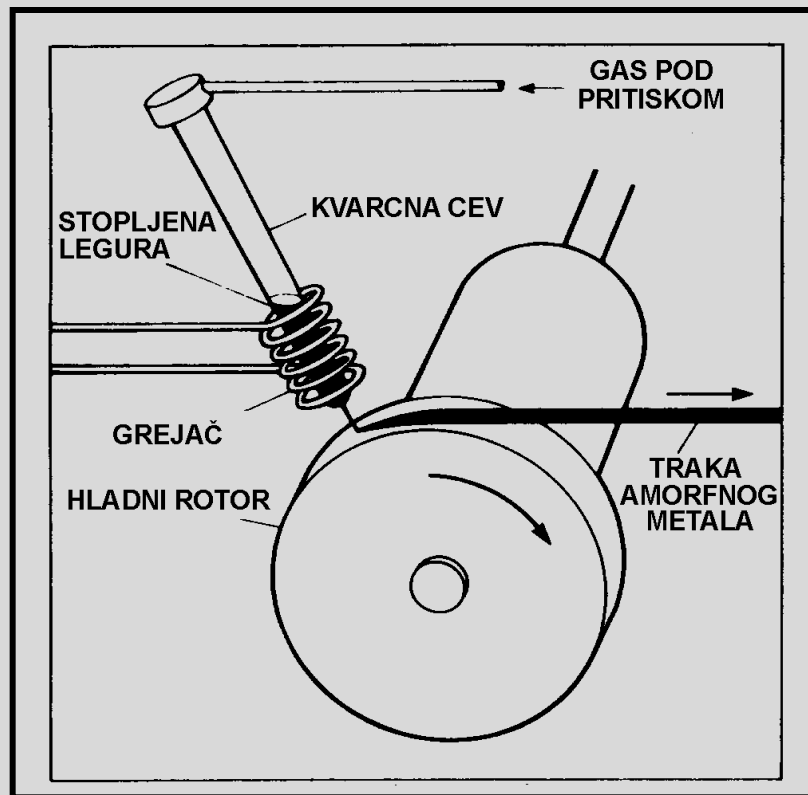


*Uzorak postavljen
za merenje
mikrtvrdoće*



*Merenje
mikrotrvrdoće*

Ispitivane amorfne legure $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$
 $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$, $\text{Fe}_{75}\text{Ni}_2\text{Si}_8\text{B}_{13}\text{C}_2$ i $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{15.5}\text{B}_7$
dobijene su pomoću metode brzog hlađenja rastopa na
rotirajućem disku u obliku traka širine 2,5 cm i debljine 35
 μm .



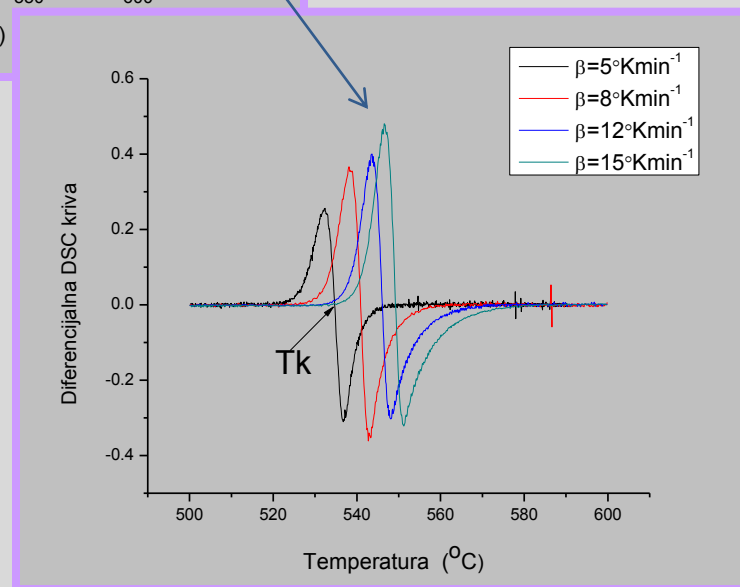
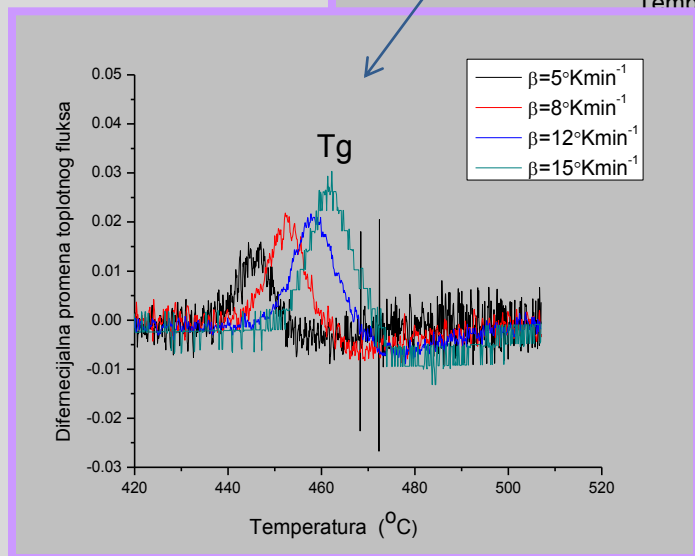
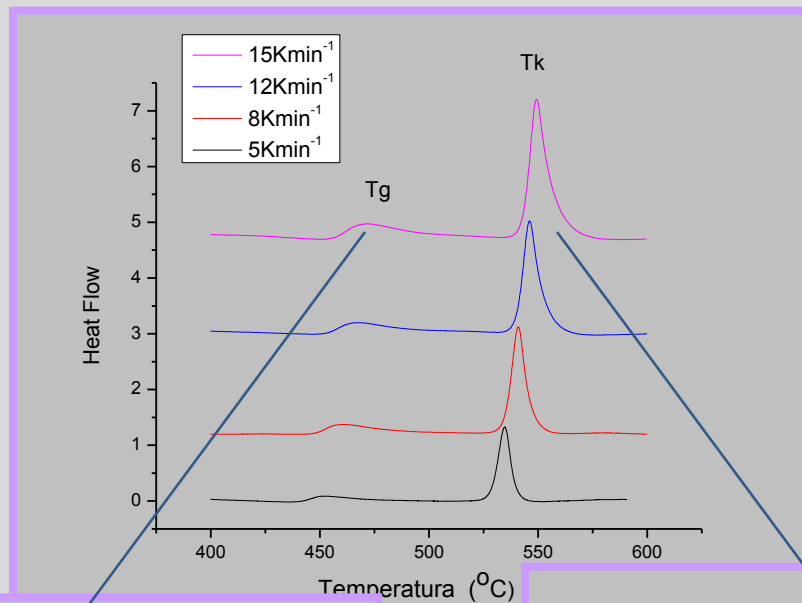
Za ispitivanje termički indukovanih strukturnih promena, uzorci polazne legure su zatapani u kvarcne cevi pod tehničkim vakuumom i izotermalno zagrevani tokom određenog vremenskog perioda (30 minuta do 24 časa).



Uzorak

Amorfna $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$ legura

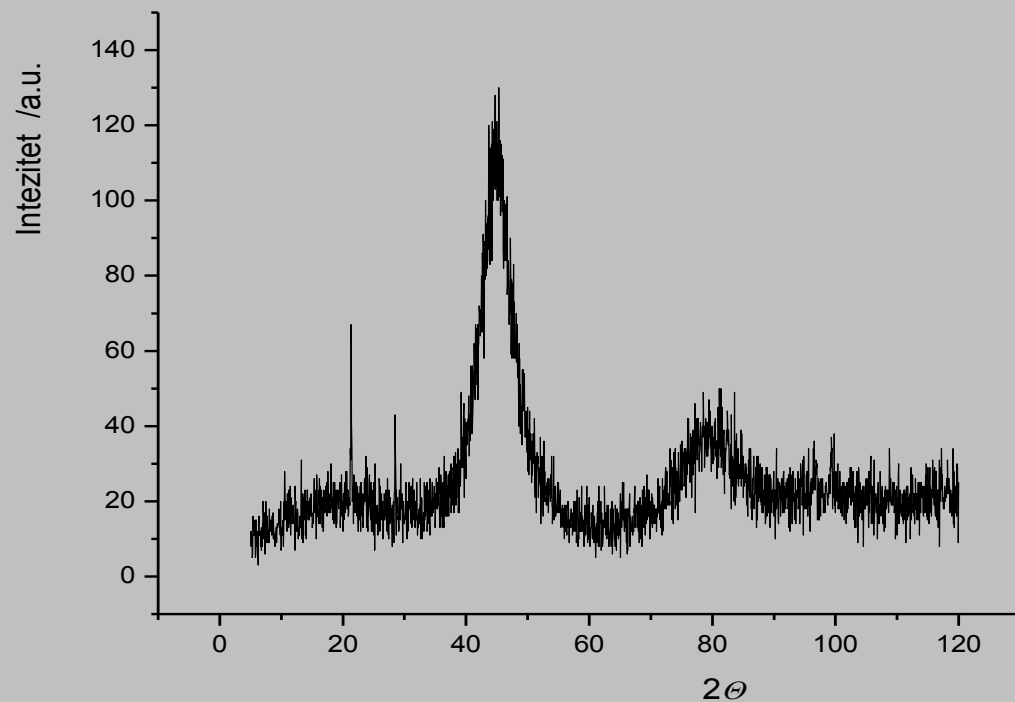
Termička stabilnost



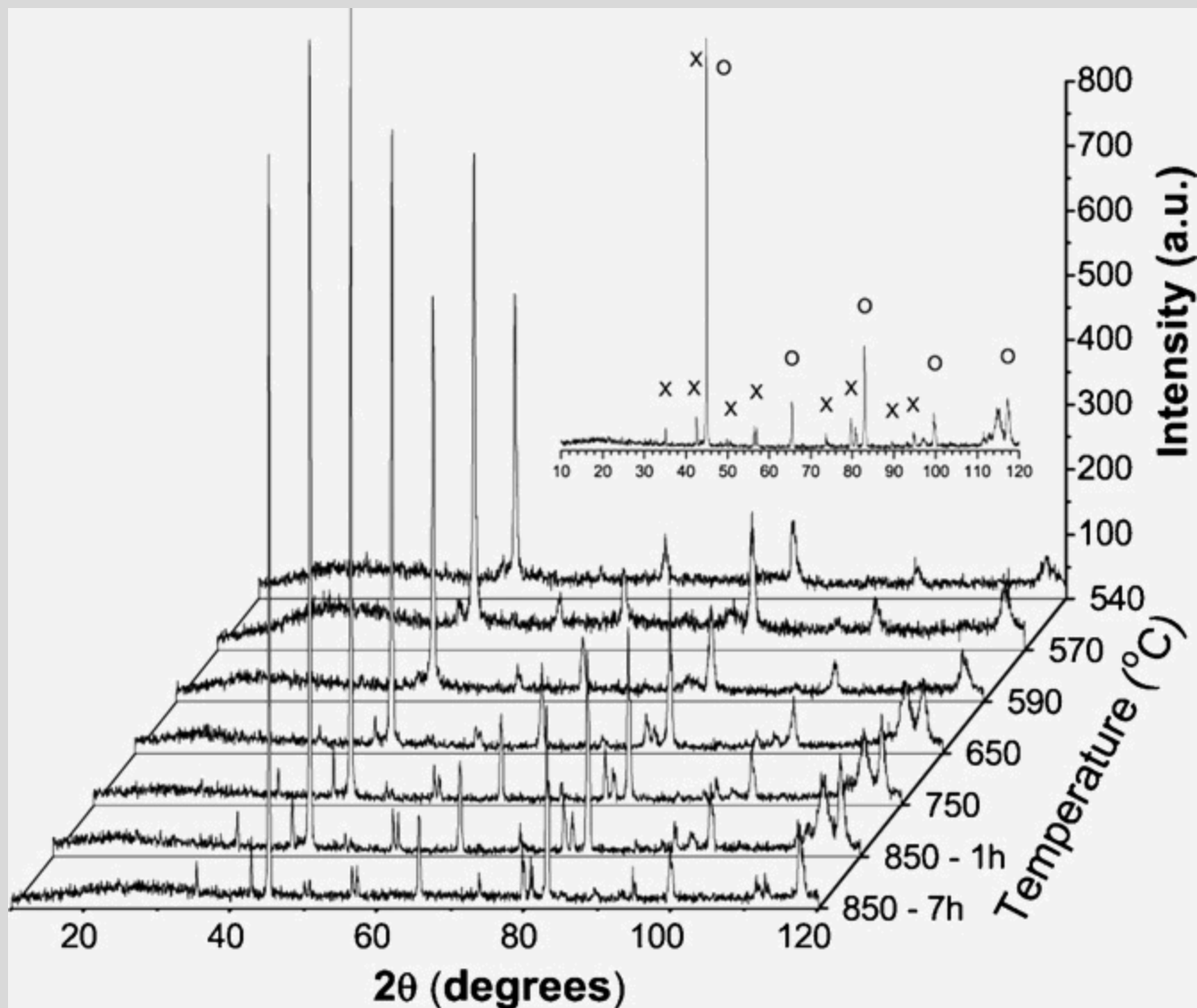
Kinetički parametri faznih transformacija

	Staklasti prelaz			Kristalizacija		
β K/min	T_g °C	E_a kJ/mol	R	T_k °C	E_a kJ/mol	R
5	445,8	297±4	0.999	534,8	410±10	0.998
8	452,3	(Kissinger)	(Kissinger)	540,8	(Kissinger)	(Kissinger)
12	458,3	294±4	0.999	545,8	403±10	0,998
15	461,3	(Ozawa)	(Ozawa)	549,3	(Ozawa)	(Ozawa)

Struktura i njene transformacije



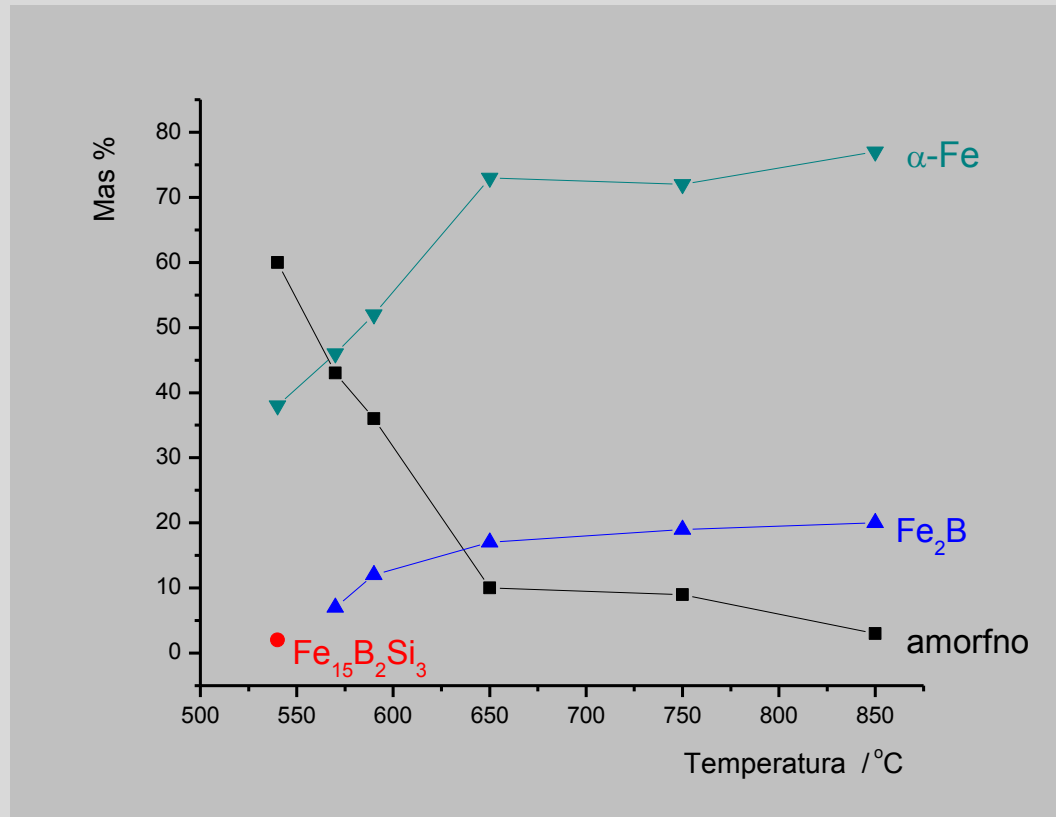
Difraktogram X-zraka polazne legure



Difraktogrami X-zraka uzoraka legure odgrevanih na raznim temperaturama

Kristalna simetrija, prostorna grupa (Hermann-Mauguin) i parametri rešetke nađenih faza u iskristalisanoj leguri

Faza	Prostorna grupa	Kristalni sistem	Parametri rešetke [Å]	
			a	c
α -Fe [ICDD-PDF 00-036-4899]	Im-3m	Kubni	2.867	-
Fe_2B [ICDD-PDF 00-036-1332]	I4/mcm	Tetragonalni	5.110	4.249
$\text{Fe}_{15}\text{B}_2\text{Si}_3$ [ICDD-PDF 00-047-1629]	I-4	Tetragonalni	8.677	4.307

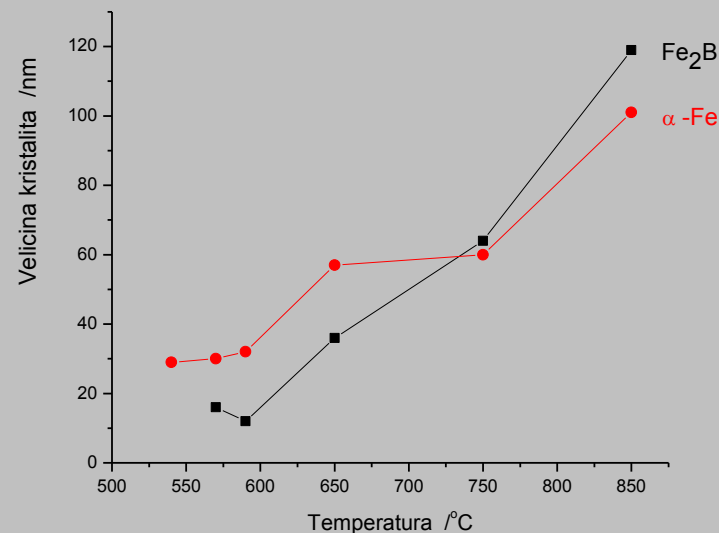


Maseni udeli nađenih kristalnih faza određeni metodom Rietveld-ovog utančavanja u funkciji temperature odgrevanja uzorka.

Veličina kristalita nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

Temperatura odgrevanja [°C]	Vreme odgrevanja [čas]	Fe ₂ B	α -Fe
		D_{hkl} [nm]	D_{hkl} [nm]
540	1	-	29
570	1	16	30
590	1	12	32
650	1	36	57
750	1	64	60
850	1	119	101
850	7	178	123

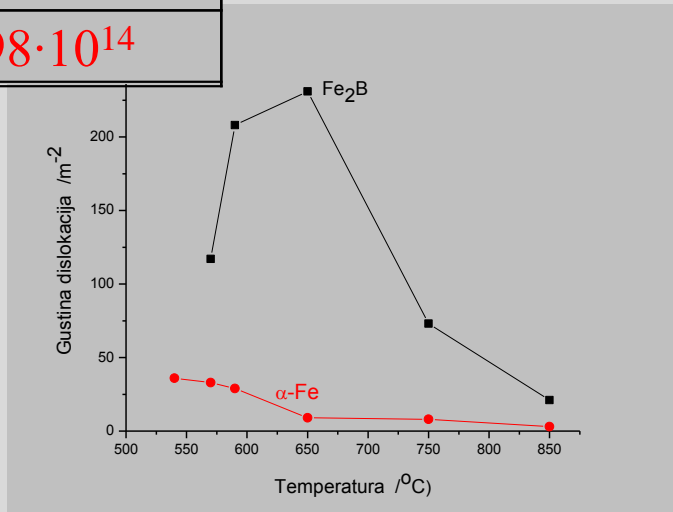
$$D_{hkl} = \frac{k\lambda}{\psi \cos \theta}$$

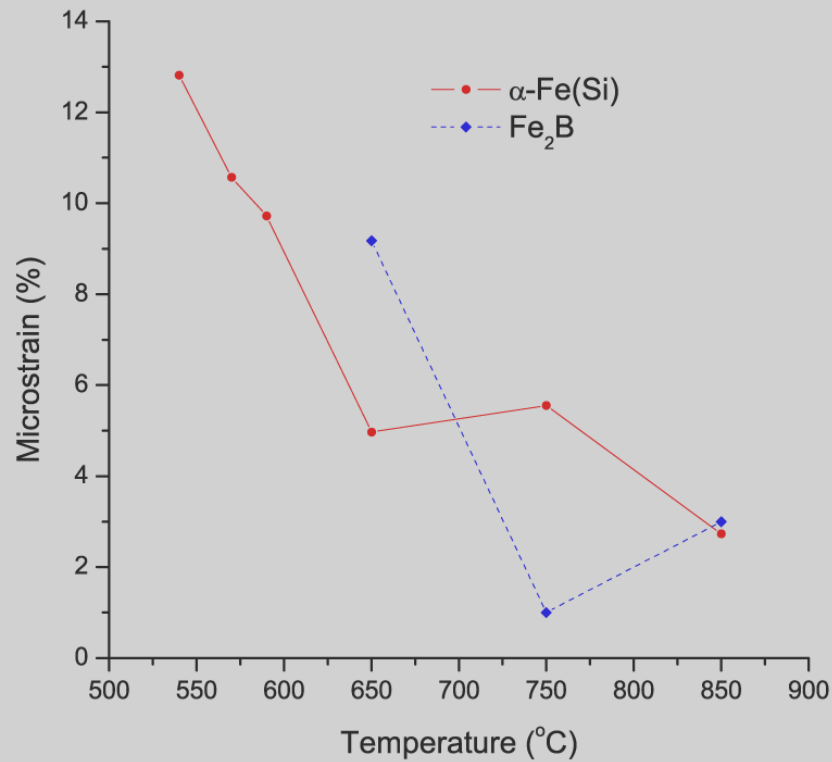


Gustina dislokacija nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

Temperature [°C]	Vreme odgrevanja [čas]	Fe ₂ B	α-Fe
		ρ_{hkl}	ρ_{hkl}
		[m ⁻²]	[m ⁻²]
540	1	-	3.57·10 ¹⁵
570	1	1.17·10 ¹⁶	3.33·10 ¹⁵
590	1	2.08·10 ¹⁶	2.93·10 ¹⁵
650	1	2.31·10 ¹⁵	9.23·10 ¹⁴
750	1	7.32·10 ¹⁴	8.33·10 ¹⁴
850	1	2.12·10 ¹⁴	2.94·10 ¹⁴
850	7	9.47·10 ¹³	1.98·10 ¹⁴

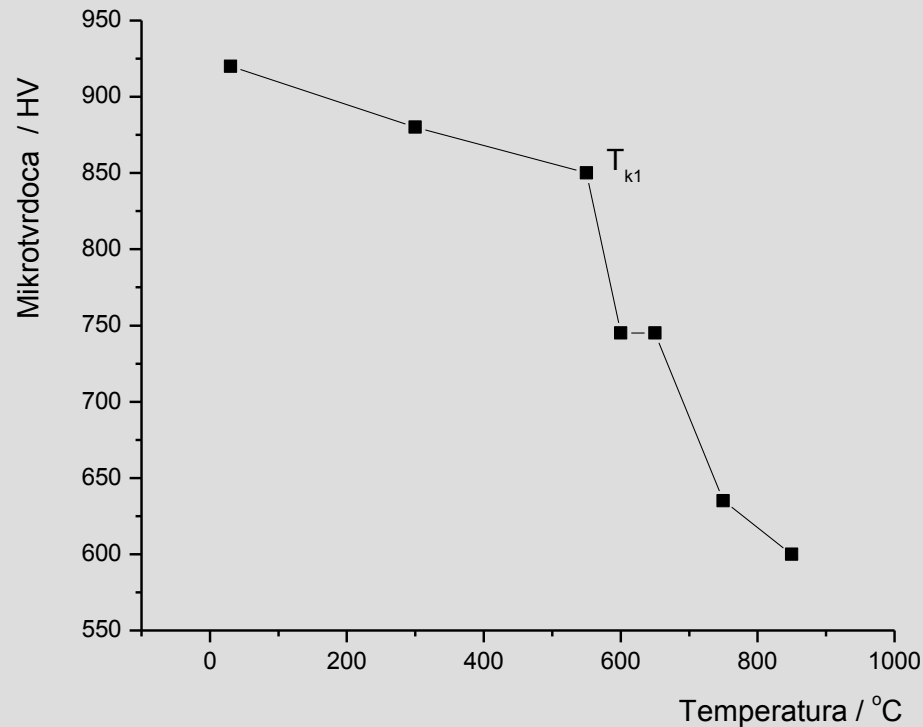
$$\rho_{hkl} = \frac{3}{D_{hkl}^2}$$





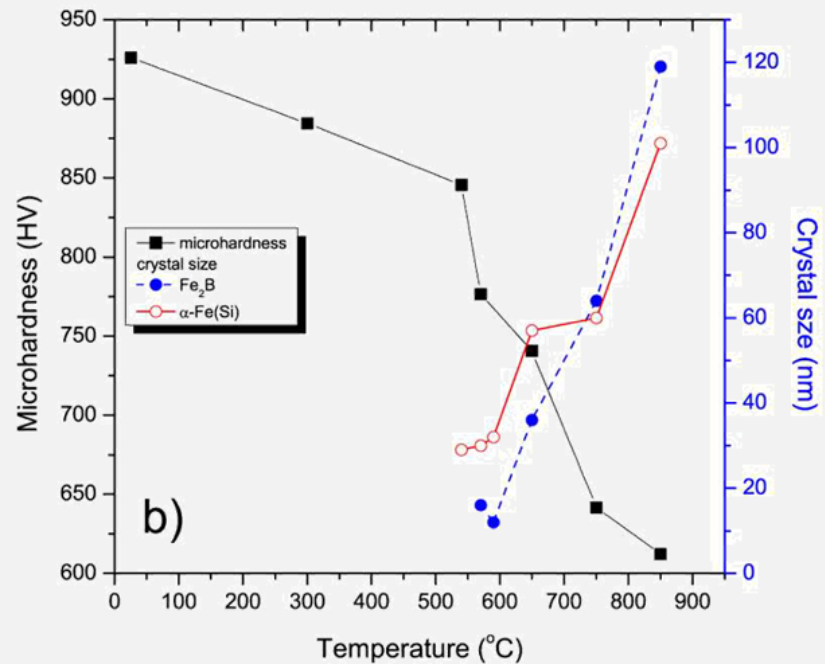
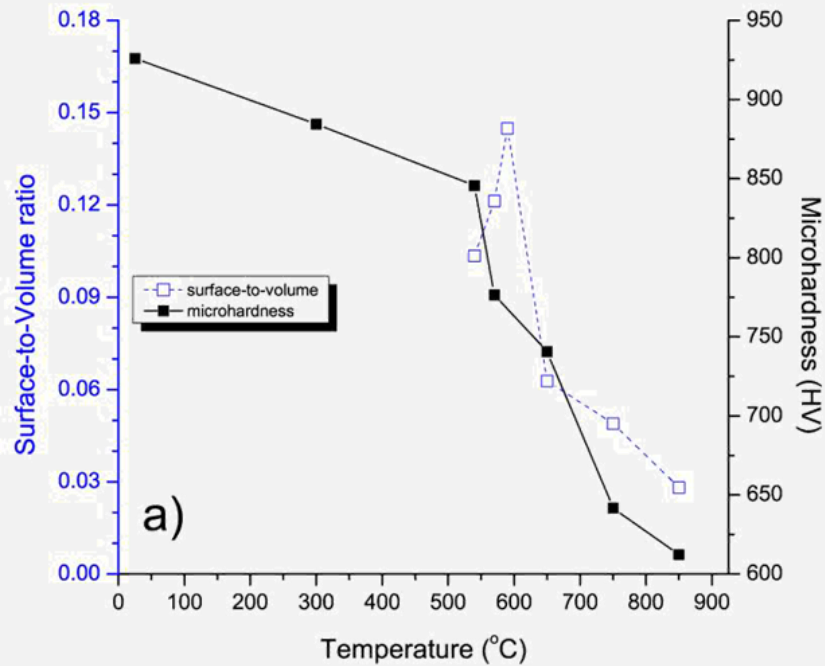
Promena mikronaprezanja sa temperaturom odgrevanja uzoraka legure.

Uticaj strukturnih promena na mikrotvrdoću

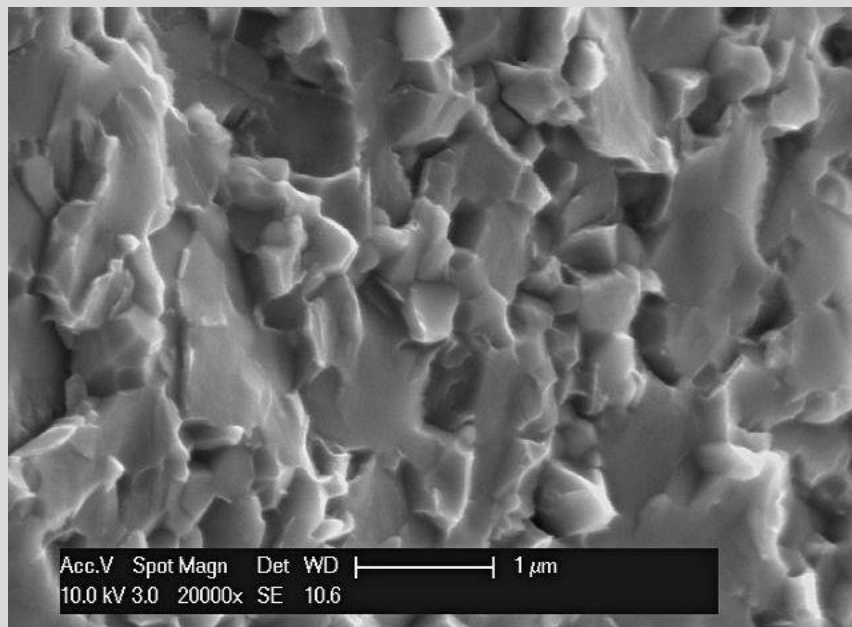


Temperaturska zavisnost mikrotvrdoće uzoraka legure odgrevanih na raznim temperaturama.

Uticaj mikrostrukturnih parametara na mikrotvrdoću odgrevane legure

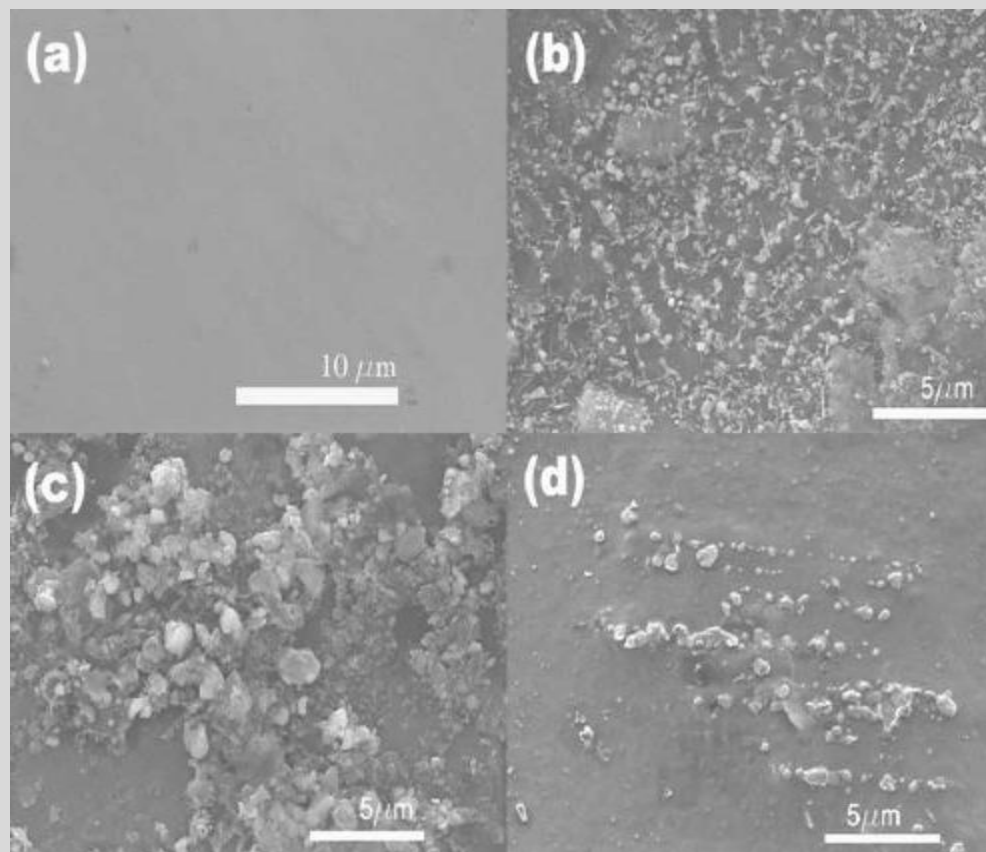


Morfologija poprečnog preseka



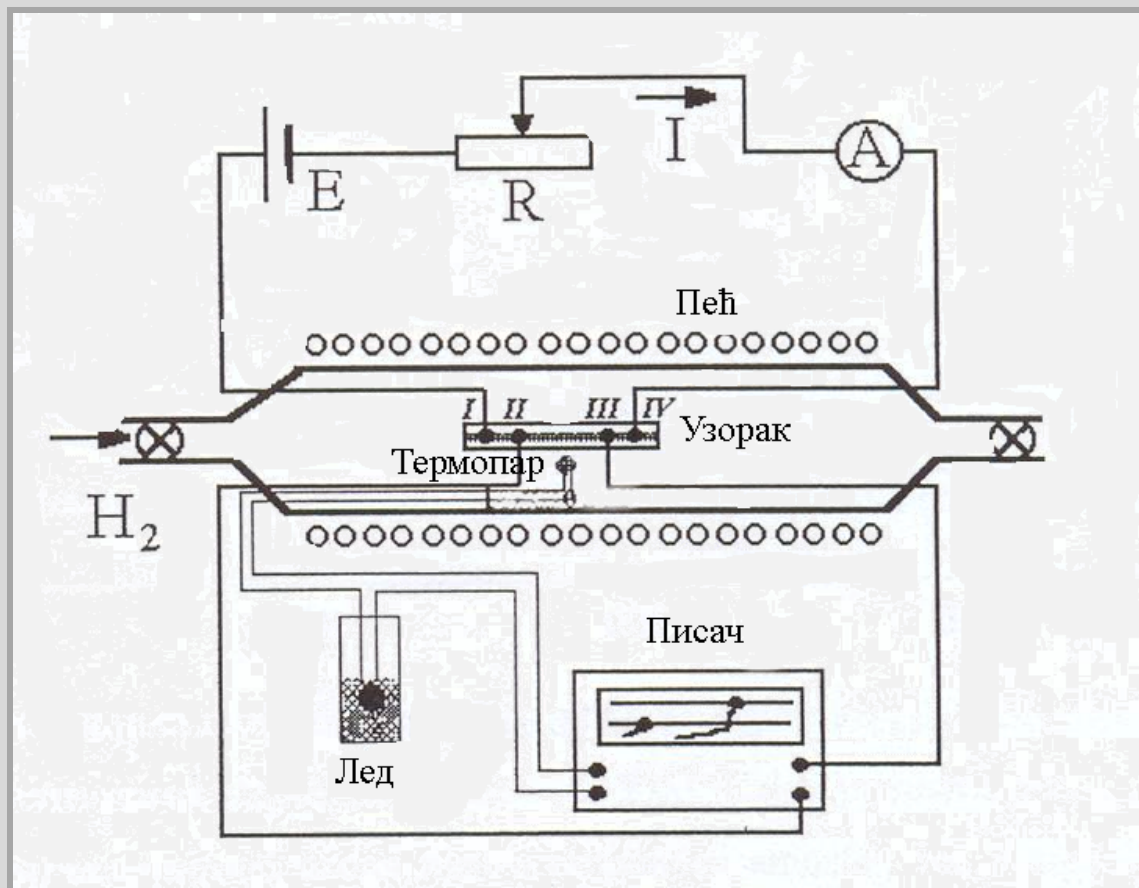
SEM micrografija poprečnog preseka uzorka legure posle odgrevanja na 850 °C

Morfologija površine

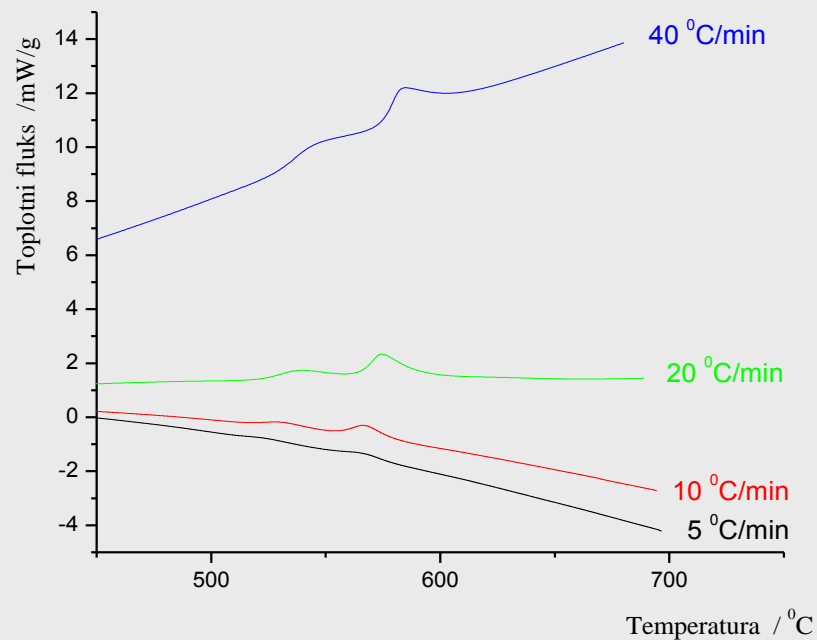


SEM mikrografije polazne legure (a) i uzoraka legure odgrevanih na (b) 650°C, (c) 750°C, (d) 850°C)

Šematski prikaz uređaja za merenje električne otpornosti u funkciji temperature



Kristalizacija

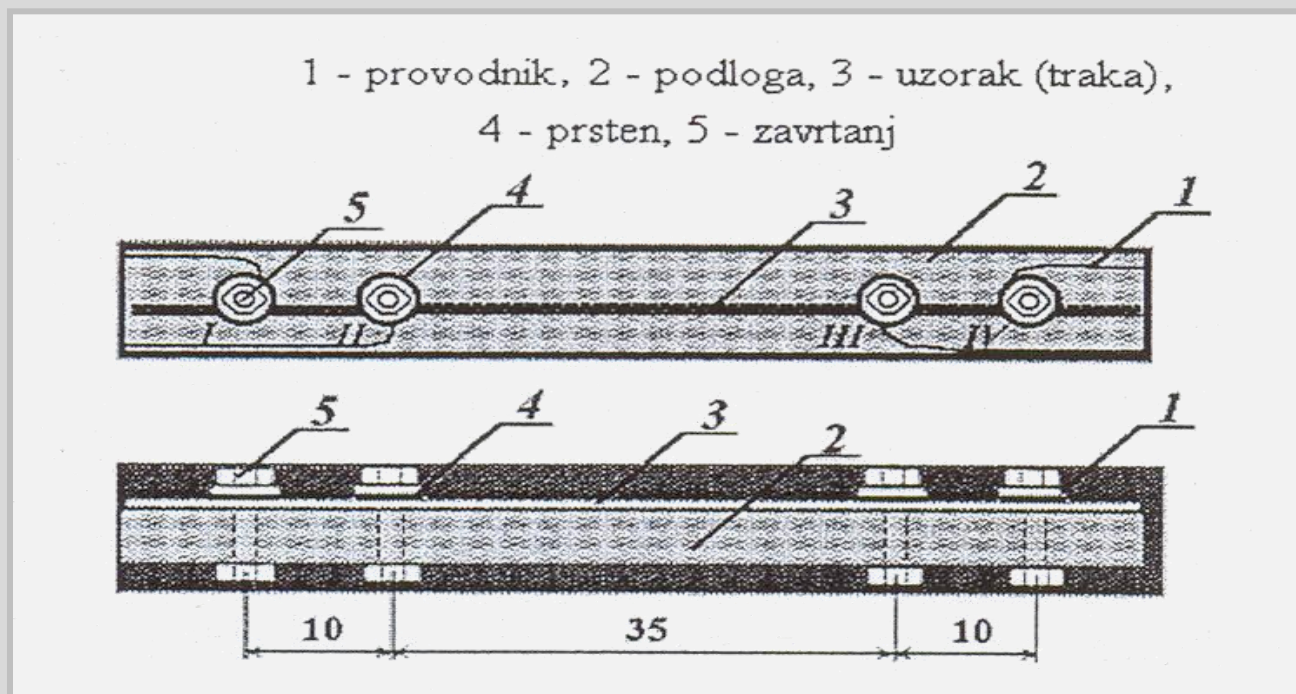


Kristalizacioni pikovi pri raznim brzinama zagrevanja u protočnoj struji azota.

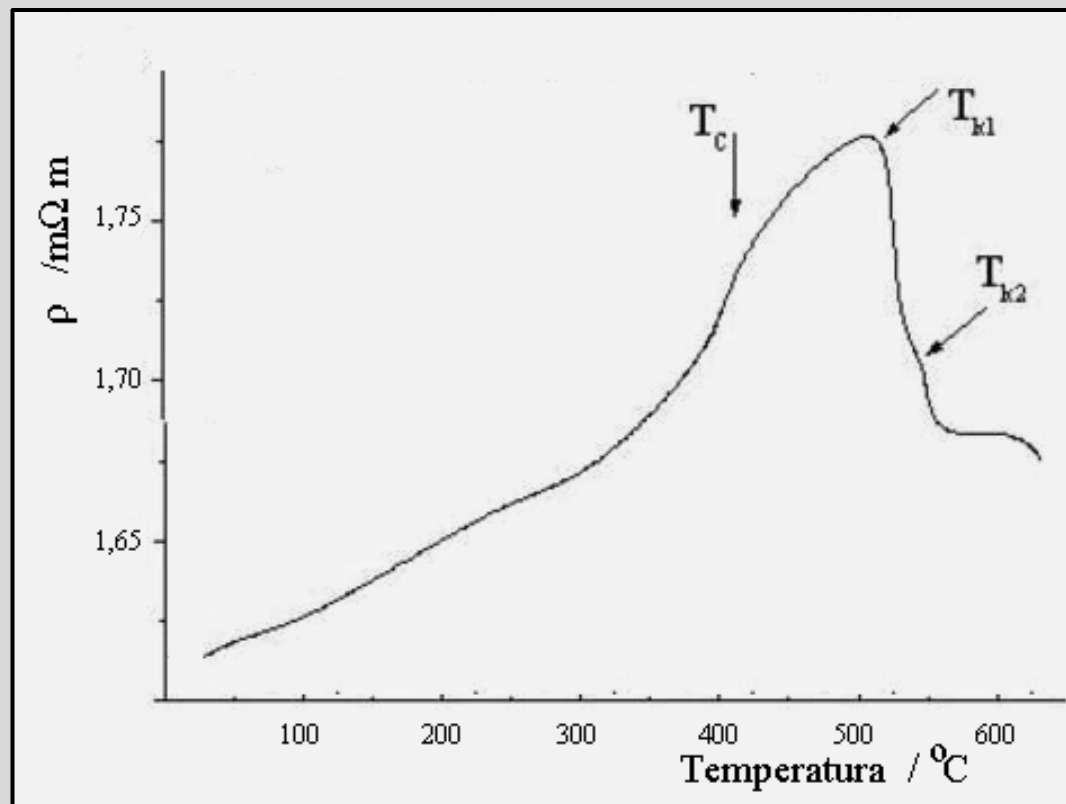
Kinetički parametri kristalizacije (prividna energija aktivacije, frekvencijski faktor, konstanta brzine) određeni po Kissinger-ovoj i Ozawa-inoj metodi

	Prvi stupanj			Drugi stupanj		
β	T_p^1	E_a^1	A^1	T_p^2	E_a^2	A^2
K/min	K	kJ/mol	min ⁻¹	K	kJ/mol	min ⁻¹
5	796,5	486,3±9 (Kissinger)	53,62 10 ²¹ (Kissinger)	-	439,3±8 (Kissinger)	4,7 10 ²⁷ (Kissinger)
10	802,9	500,0±10 (Ozawa)	1,8 10 ³⁸ (Ozawa)	839,5	460,8±8 (Ozawa)	2,6 10 ²⁴ (Ozawa)
20	811,9			847,9		
40	819,0			857,5		

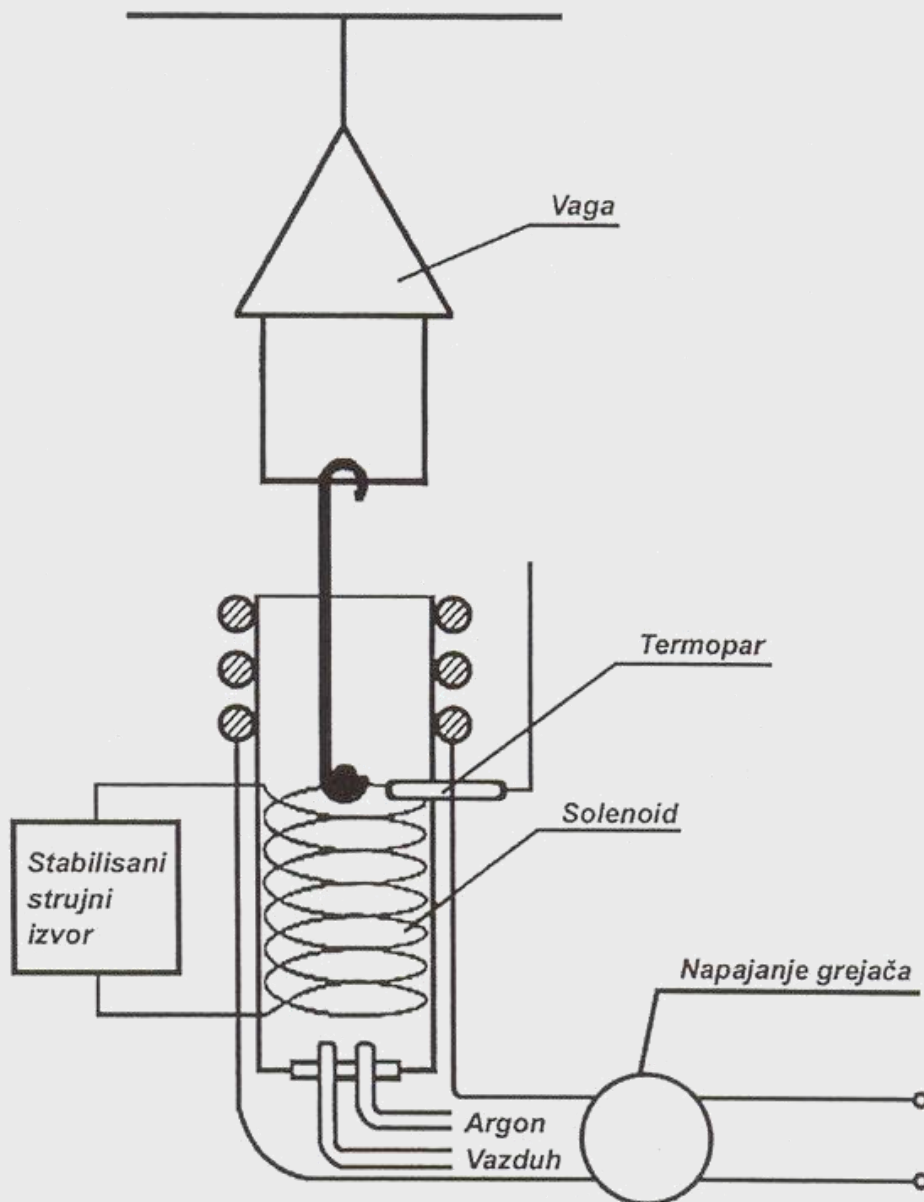
Položaj uzorka tokom merenja električne otpornosti
(metod četiri tačke)



Uticaj strukturnih promena na električna svojstva

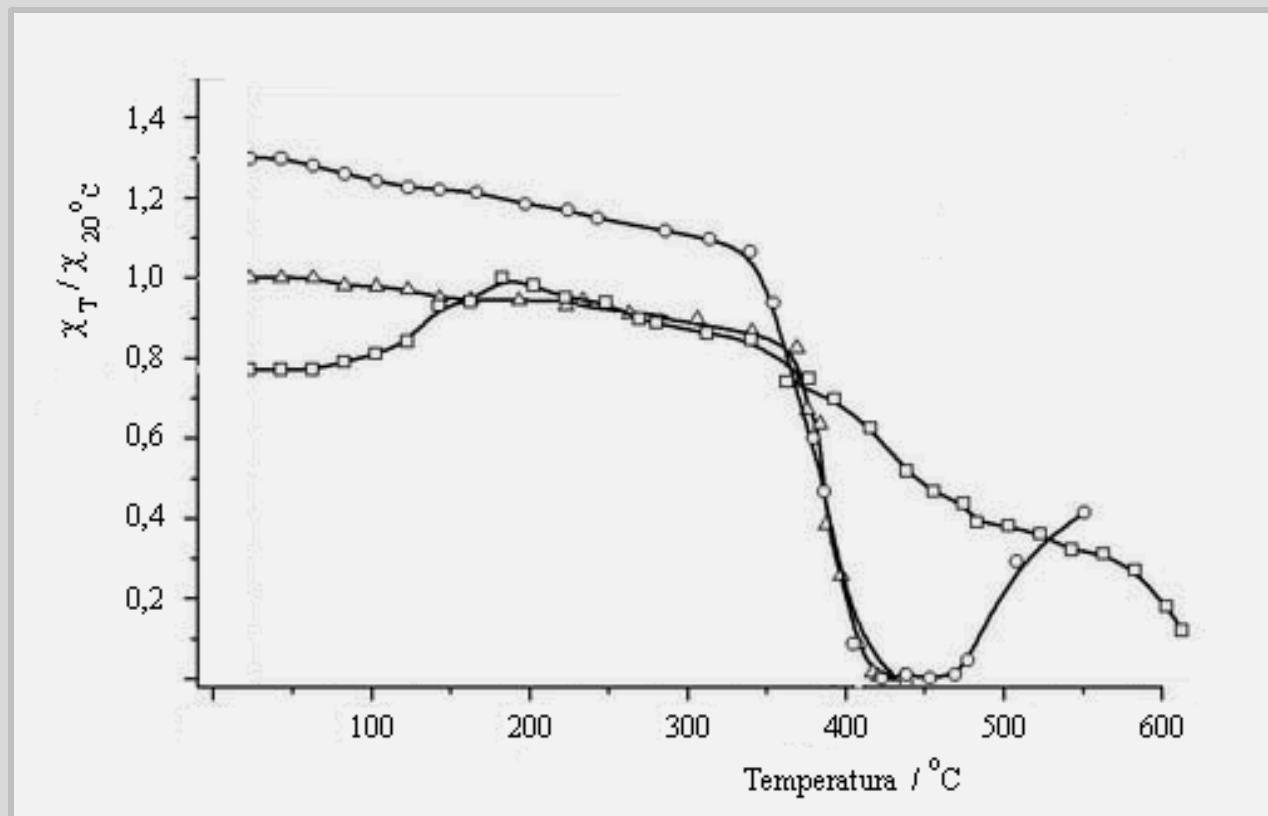


Promena električne otpornosti
sa temperaturom za polaznu
amorfnu leguru



Šemtski prikaz uređaja
za određivanje
relativne promene
magnetne
susceptibilnosti po
modifikovanoj
Faraday-evoj metodi

Uticaj strukturnih promena na magnetna svojstva



$T_c = 430^\circ\text{C}$

Temperaturska zavisnost relativne magnetne susceptibilnosti za tri ciklusa zagrevanja legure do različitih temperatura.

Literatura

Kalezić-Glišović, L. Novaković, A. Maričić, D. M. Minić and N. Mitrović, *Correlation Between Processes of Structural Relaxation and Crystallization and Magnetic Susceptibility Relative Change of the $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$ Amorphous Alloy*, Materials Science and Engineering B, 131 (1-3) (2006) 45-48.

D. M. Minić, A. Gavrilović, P. Angerer, D. G. Minić, A. Maričić, *Thermal Stability and Crystallization of $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$* , Journal of Alloys and Compounds, 482(2009) 502-507.

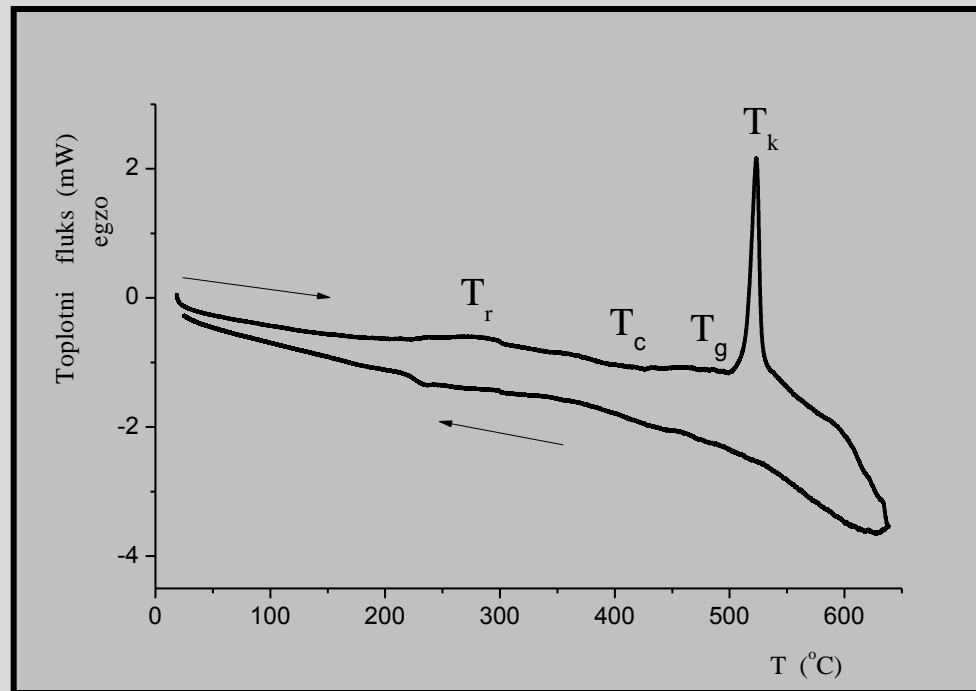
• D.M. Minić, V. Blagojević, D.G. Minić, A. Gavrilović, L. Rafailović, The influence of thermally induced structural transformations on hardness in $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$ amorphous alloy, Journal of Alloys and Compounds, 509(2011) 8350-8355.

• Vladimir A. Blagojević, Dušan M. Minić, Milica Vasić, Dragica M. Minić, *Effect of Thermal Treatment on Structural and Physical Properties of $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$ Amorphous Alloy*, Mater. Phys.Chem, 142(1)(2013)207-212

• Milica Vasić, Dušan M. Minić, Vladimir A. Blagojević, Dragica M. Minić, *Mechanism of Thermal Stabilization of $\text{Fe}_{89.8}\text{Ni}_{1.5}\text{Si}_{5.2}\text{B}_3\text{C}_{0.5}$ Amorphous Alloy*, Thermochimica Acta 562 (2013) 35-41.

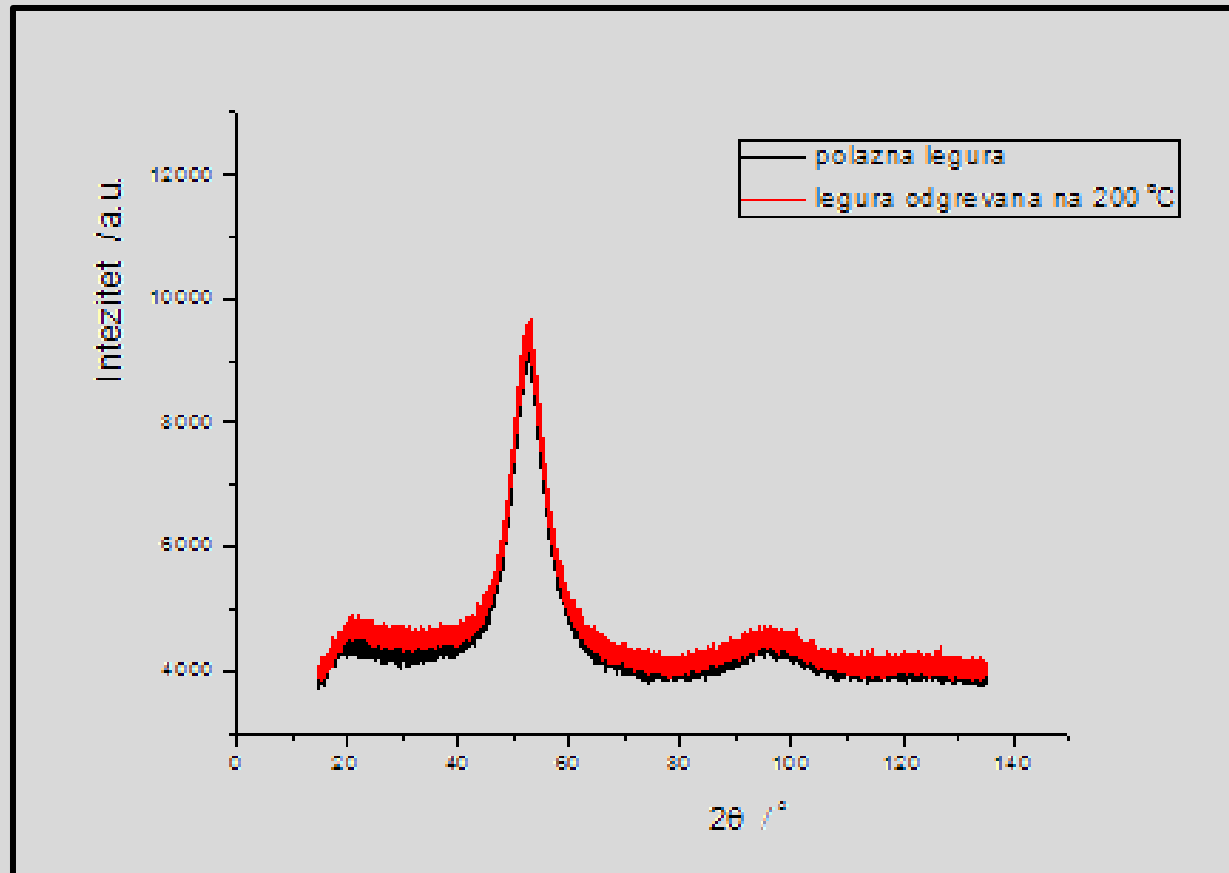
Amorfna $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ legura

Termička stabilnost

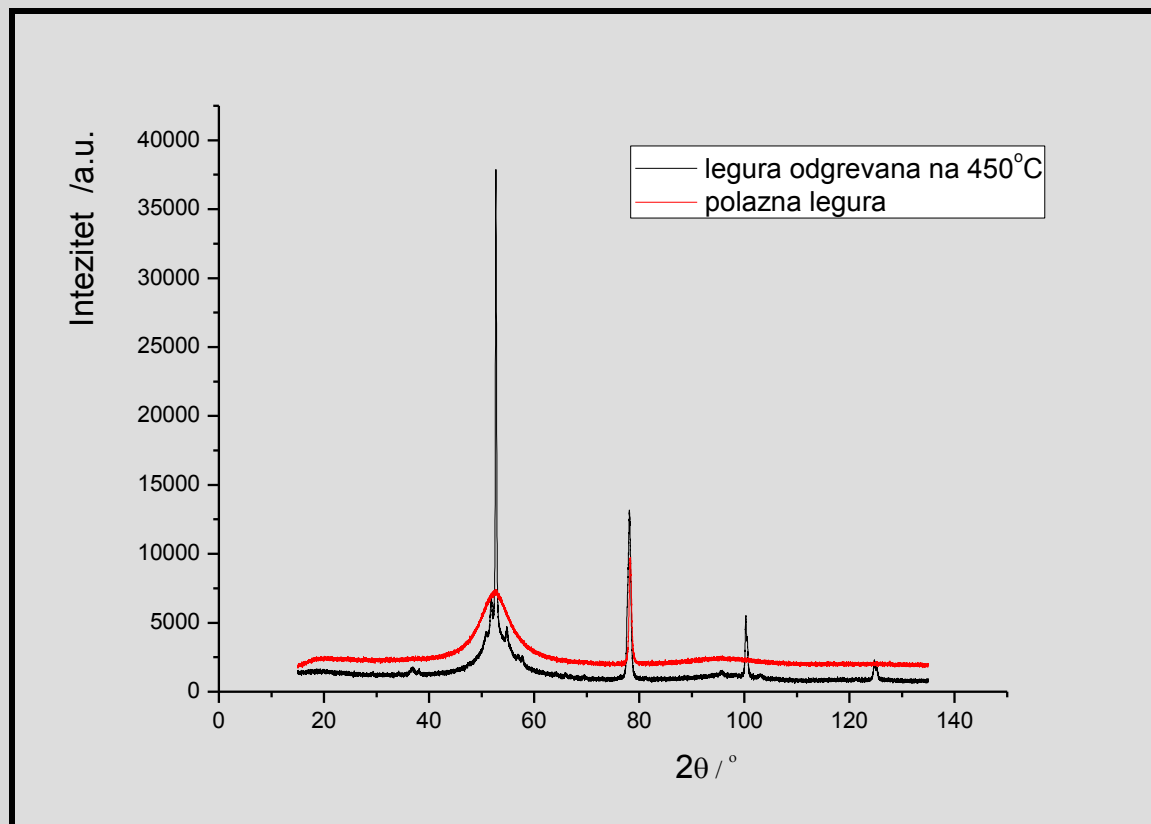


DSC termogram polazne legure u ciklusu zagrevanja i hlađenja u protočnoj struji azota pri brzini odgrevanja $10^\circ\text{C}/\text{min}$.

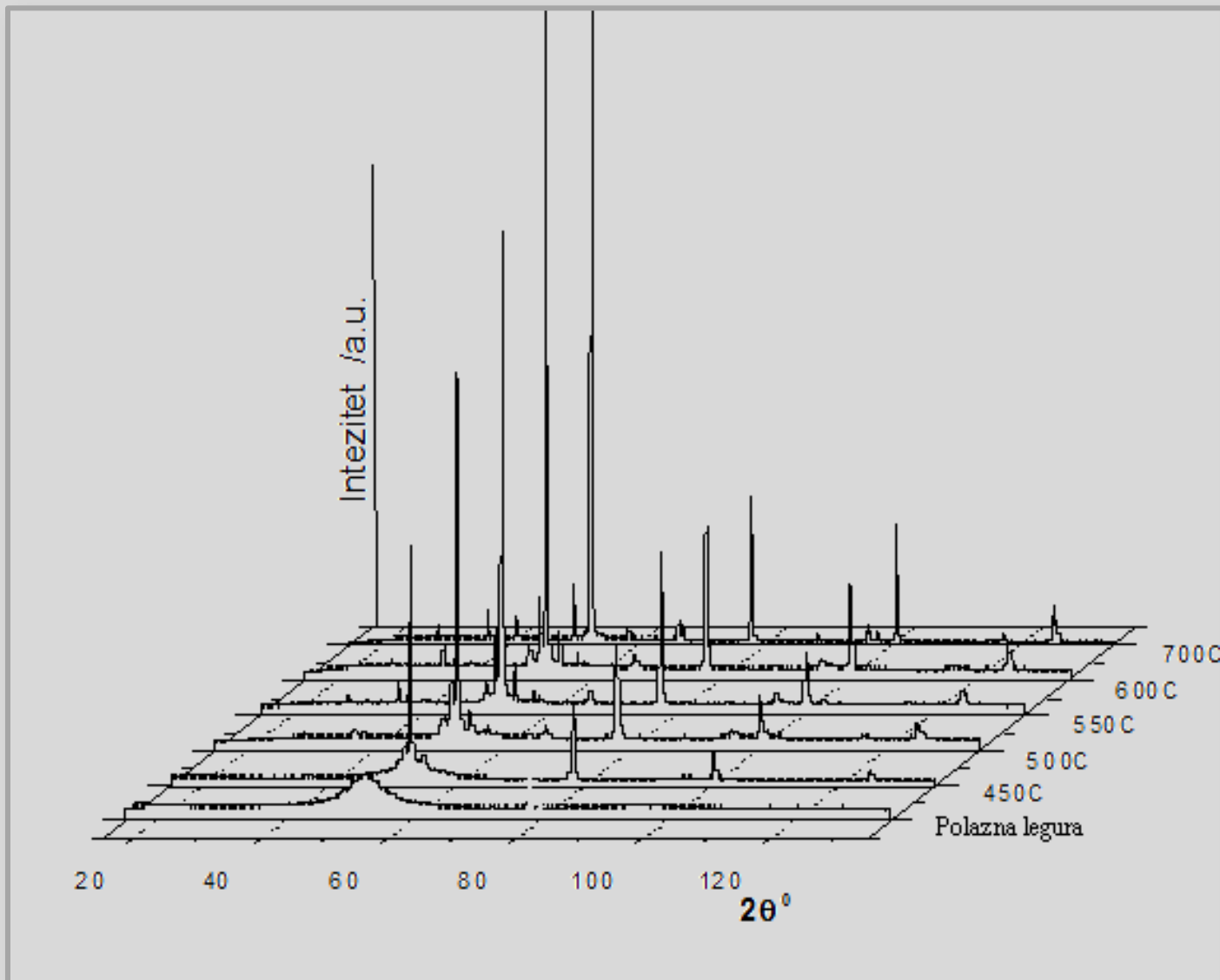
Strukturne transformacije



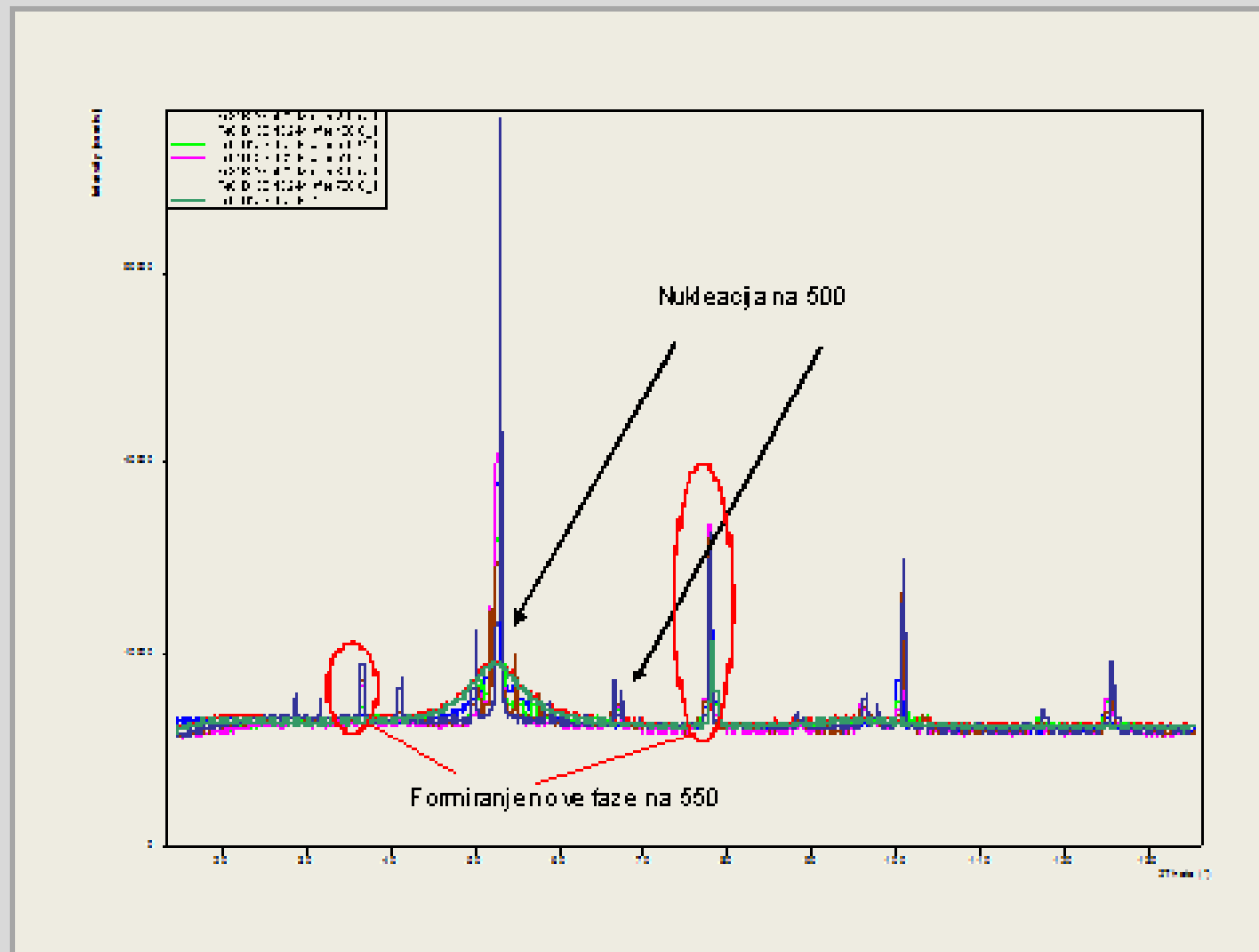
Uporedni difraktogrami X-zraka polazne legure i uzorka legure odgrevanog na 200°C.



Uporedni difraktogrami X-zraka polazne legure i uzorka legure odgrevanog na 450°C.



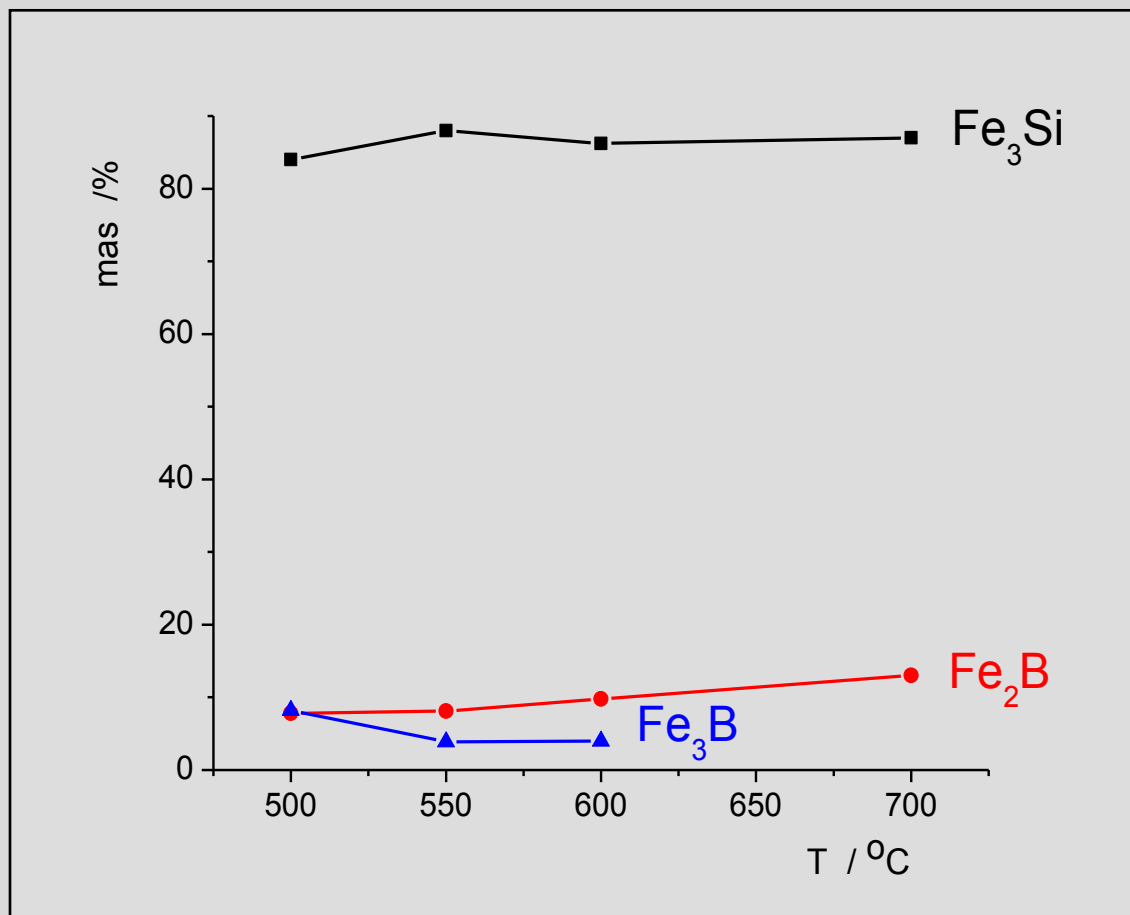
Difraktogrami X-zraka polazne legure i uzoraka legure odgrevanih na raznim temperaturama



Uporedni difraktogrami X-zraka polazne legure i uzoraka legure odgrevanih na raznim temperaturama sa izvučenim oblastima uočenih promena.

Kristalna simetrija, prostorna grupa (Hermann-Mauguin) i parametri rešetke nađenih faza u iskristalisanjoj leguri

Faza	Prostorna grupa	Kristalni sistem	Parametri rešetke [Å]	
			a	c
Fe ₃ Si [ICDD-PDF 00-042-1329]	Fm-3m	Kubni	5.662	-
Fe ₂ B [ICDD-PDF 00-036-1332]	I4/mcm	Tetragonalni	5.110	4.249
Fe ₃ B [ICDD-PDF 00-039-1316]	I-4	Tetragonalni	8.674	4.313

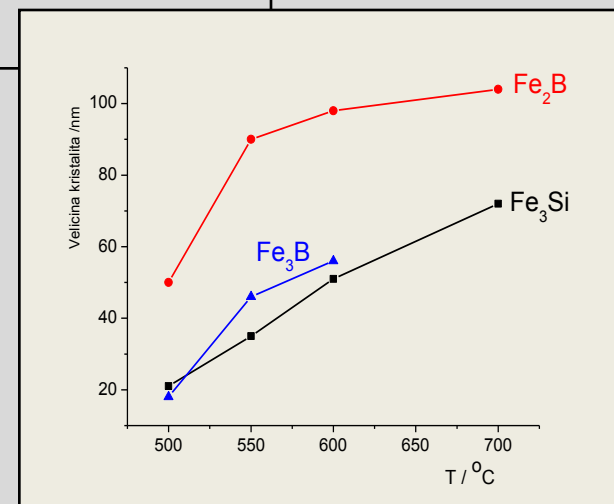


Maseni udeli nađenih kristalnih faza određeni metodom Rietveld-ovog utančavanja u funkciji temperature odgrevanja uzorka.

Veličina kristalita nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

Temperatura [°C]	Vreme zagrevanja [h]	Fe ₃ Si	Fe ₂ B	Fe ₃ B
		Veličina kristalita [nm]	Veličina kristalita [nm]	Veličina kristalita [nm]
500	0,5	22 ± 0,4	49 ± 1,4	16 ± 0,7
550	0,5	35 ± 0,7	91 ± 2,2	46 ± 1,2
600	0,5	52 ± 1,3	98 ± 2,3	57 ± 1,3
700	0,5	72 ± 1,7	108 ± 2,8	

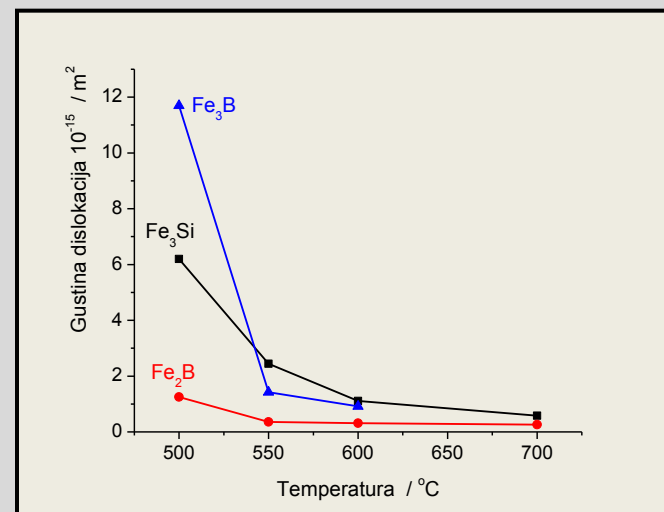
$$D_{hkl} = \frac{k\lambda}{\psi \cos \theta}$$



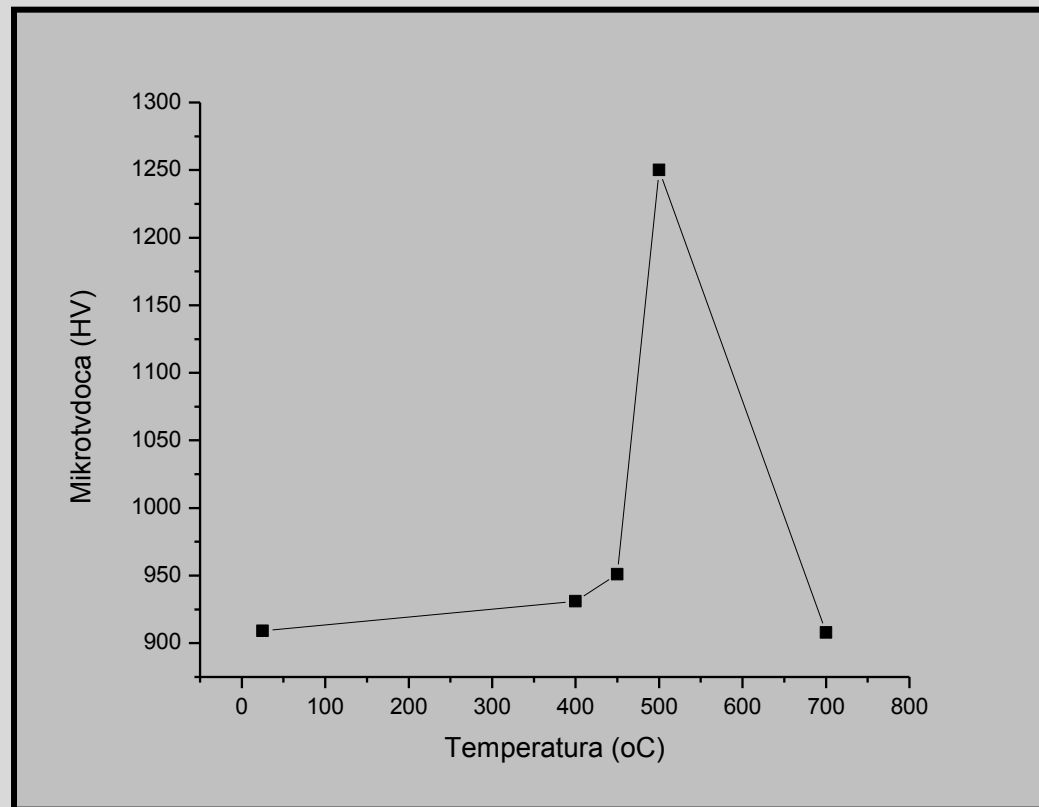
Gustina dislokacija nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

Temperatura [°C]	Vreme zagrevanja [h]	Fe ₃ Si	Fe ₂ B	Fe ₃ B
		ρ_{hkl} [m ⁻²]	ρ_{hkl} [m ⁻²]	ρ_{hkl} [m ⁻²]
500	0,5	$6,20 \cdot 10^{15}$	$1,25 \cdot 10^{15}$	$1,17 \cdot 10^{16}$
550	0,5	$2,44 \cdot 10^{15}$	$3,62 \cdot 10^{14}$	$1,42 \cdot 10^{15}$
600	0,5	$1,11 \cdot 10^{15}$	$3,12 \cdot 10^{14}$	$9,23 \cdot 10^{14}$
700	0,5	$5,79 \cdot 10^{14}$	$2,56 \cdot 10^{14}$	-

$$\rho_{hkl} = \frac{3}{D_{hkl}^2}$$

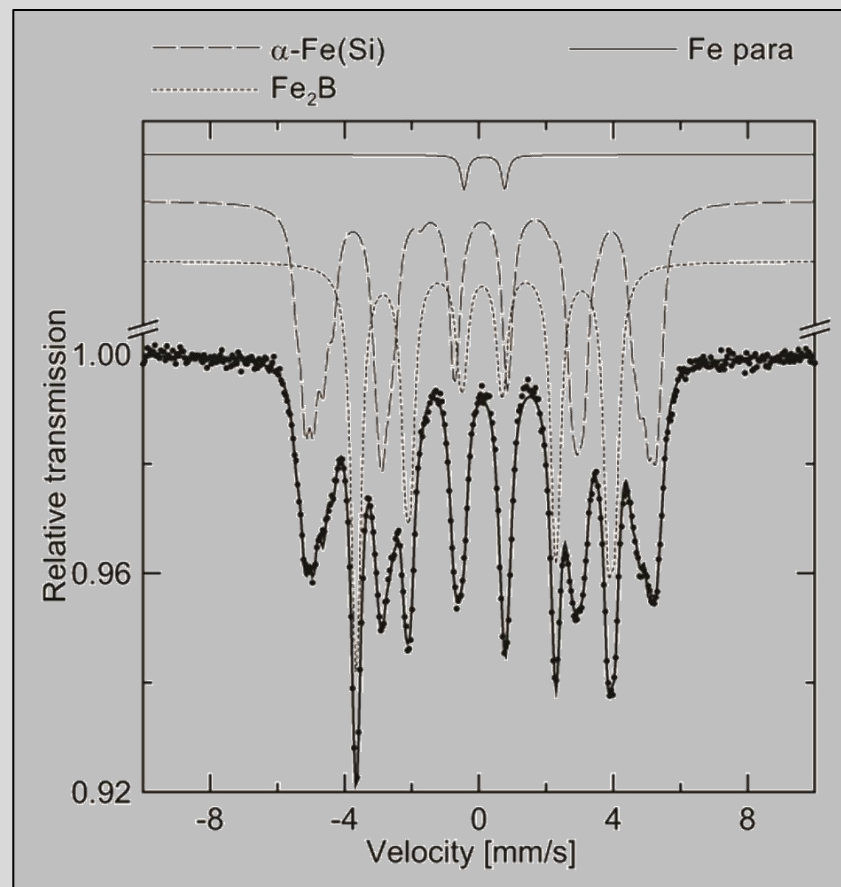
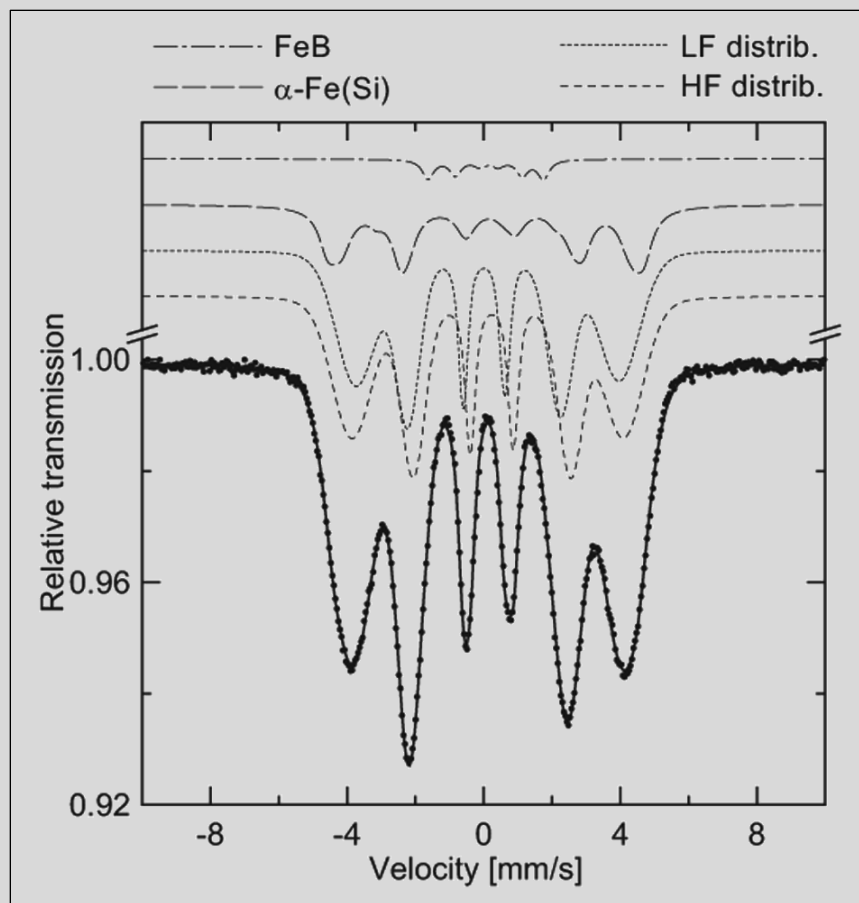


Uticaj strukturnih promena na mikrotvrdoću



Temperaturska zavisnost mikrotvrdoće uzoraka legure odgrevanih na raznim temperaturama.

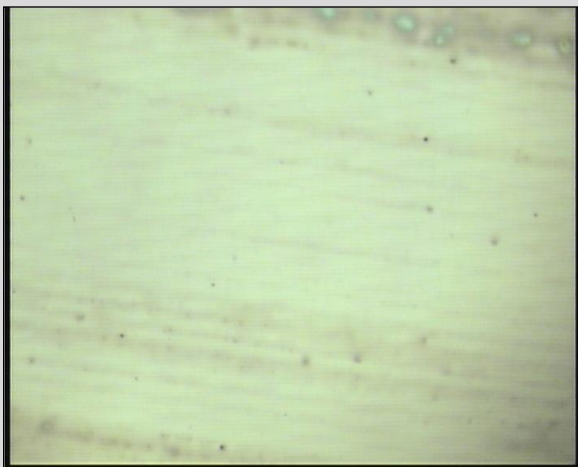
Mössbauer spektri polazne legure (levo) i posle odgrevanja na 700°C (desno), uključujući faze koje sadrže Fe. LF i HF označavaju low-field i high-field komponentu amorfne faze



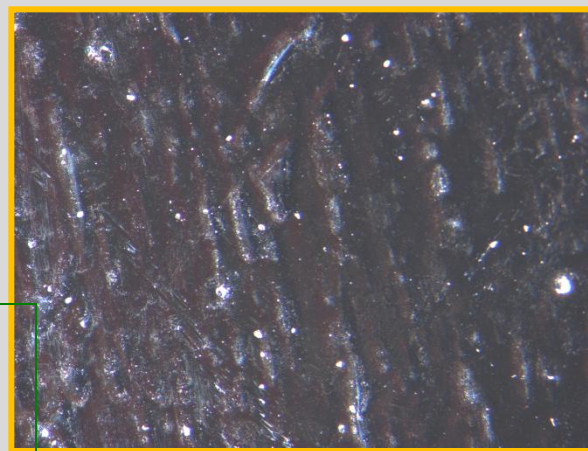
Mössbauer-tentativna fazna analiza (Mössbauer-ova distribucija Fe-atoma)

Annealing temperature	Amorphous	α -Fe(Si)	Fe ₂ B	Fe ₃ B	FeB	α -Fe	Fe para
as-prepared alloy	0.95	0.03	—	—	0.02	—	—
200 °C/30 min.	0.94	0.02	—	—	0.02	0.02	—
450 °C/30 min.	0.83	0.14	—	0.03	—	—	—
500 °C/30 min.	—	0.42	0.42	0.15	—	—	0.01
550 °C/30 min.	—	0.52	0.47	—	—	—	0.01
600 °C/30 min.	—	0.54	0.45	—	—	—	0.01
700 °C/30 min.	—	0.55	0.44	—	—	—	0.01

Morfologija površine

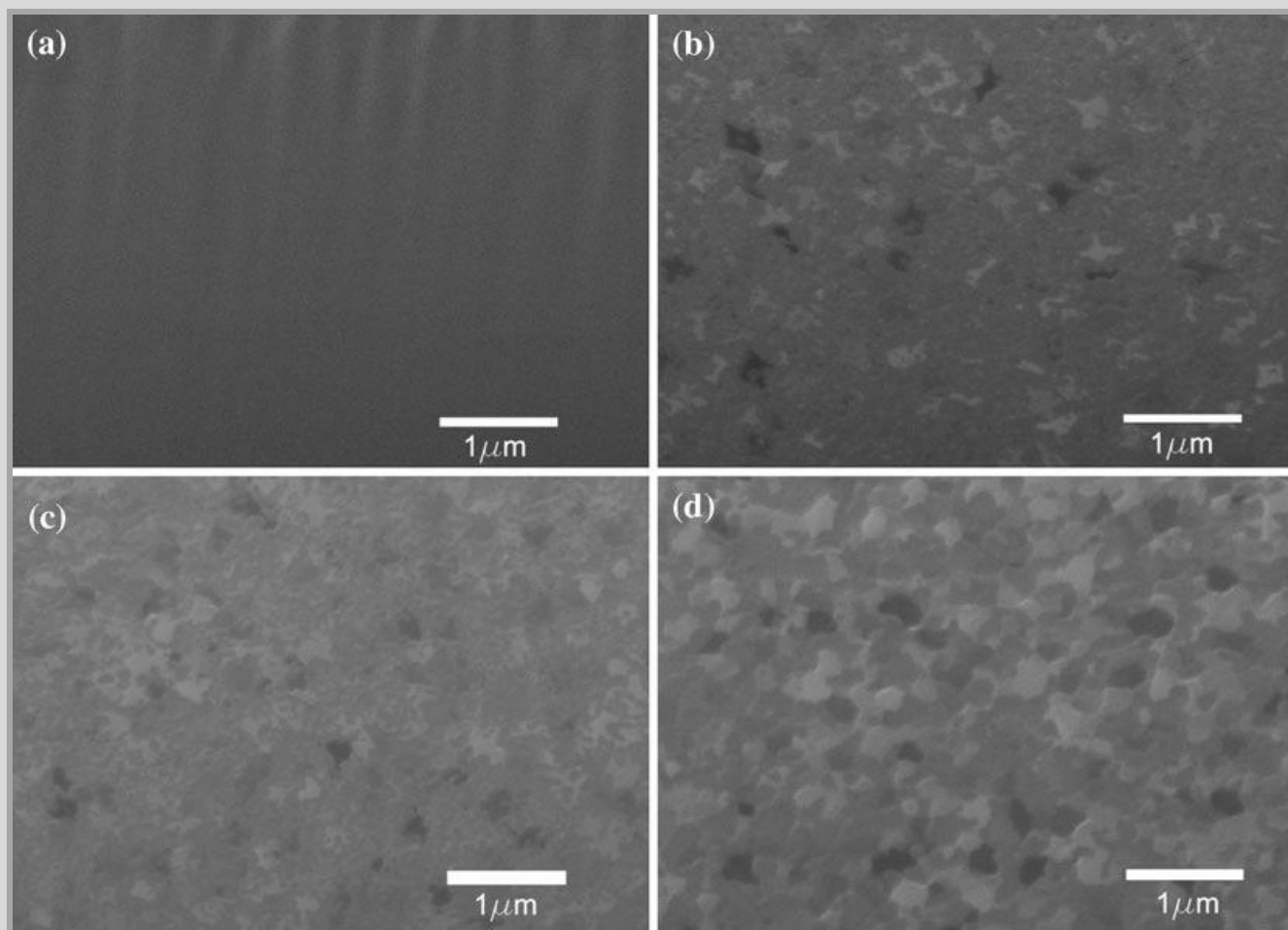


Homogena površina uzorka
polazne legure

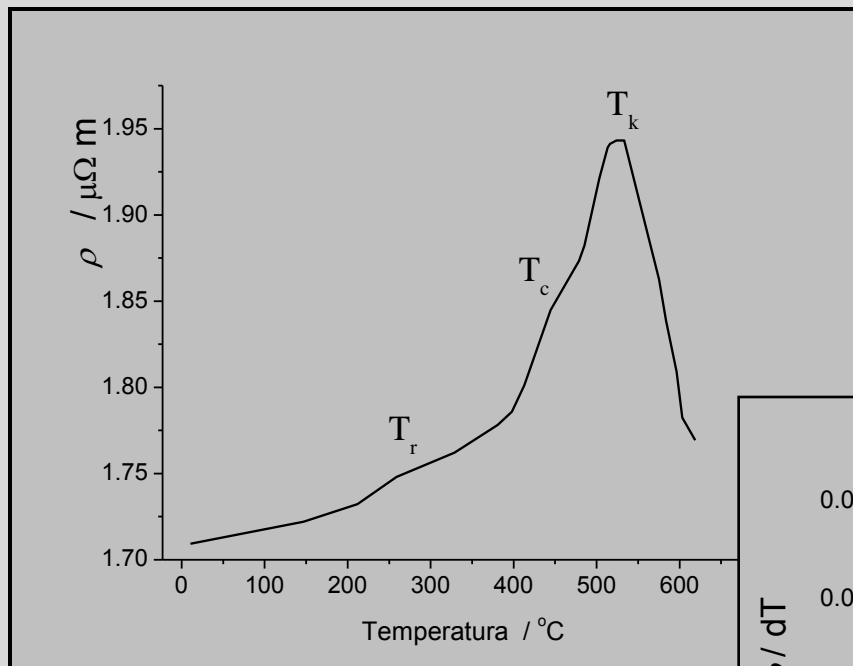


Zrnasta površina uzorka
legure odgrejane na 600°C

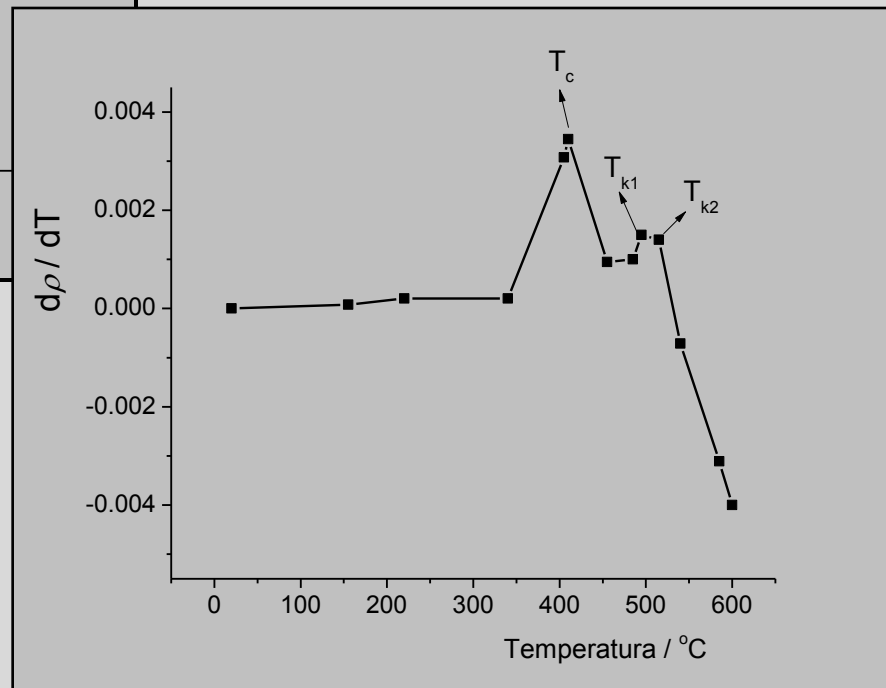
FIB-SEM amorfne legure i uzoraka legure koji su idgrevan na: (a) 473 K (200°C), (b) 773 K (500°C), (c) 873 K (600°C), (d) 973 K (700°C).

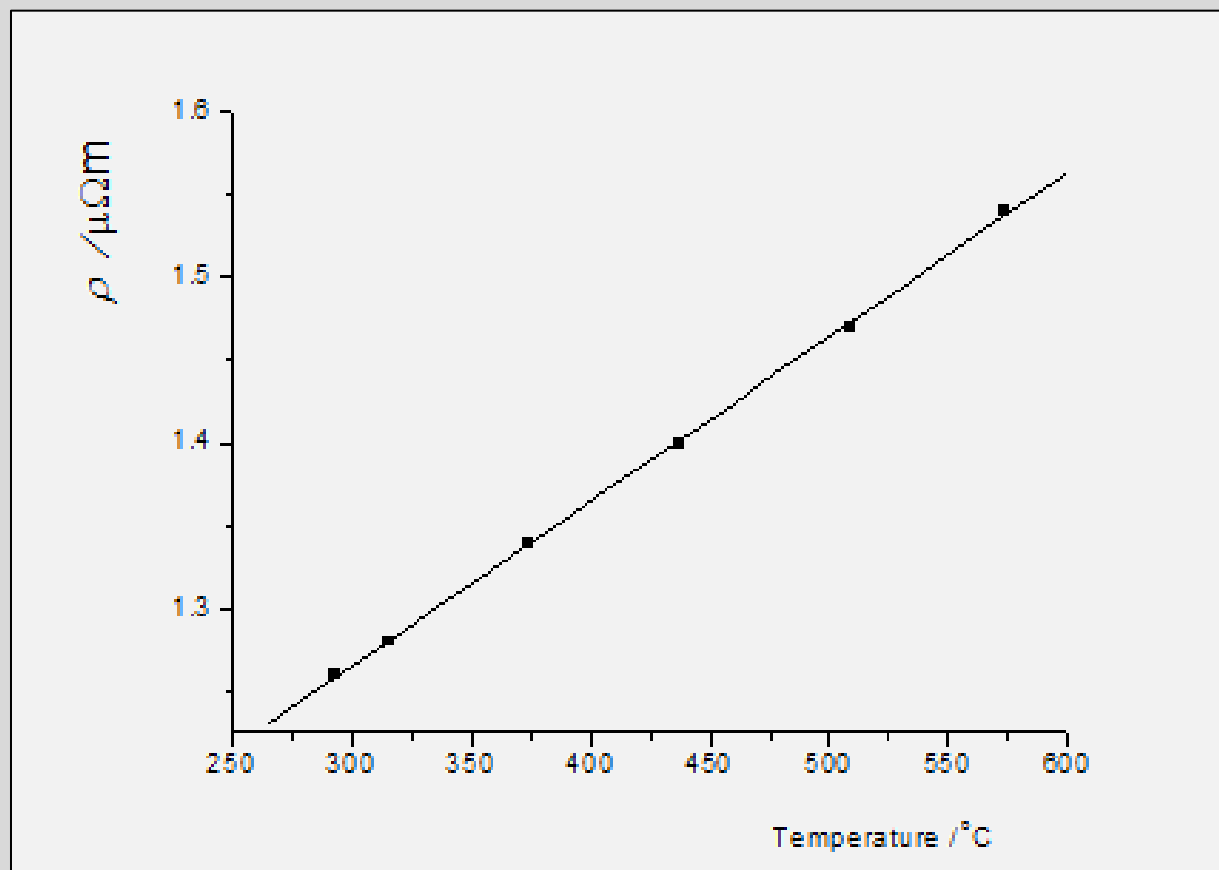


Uticaj strukturnih promena na električna svojstva



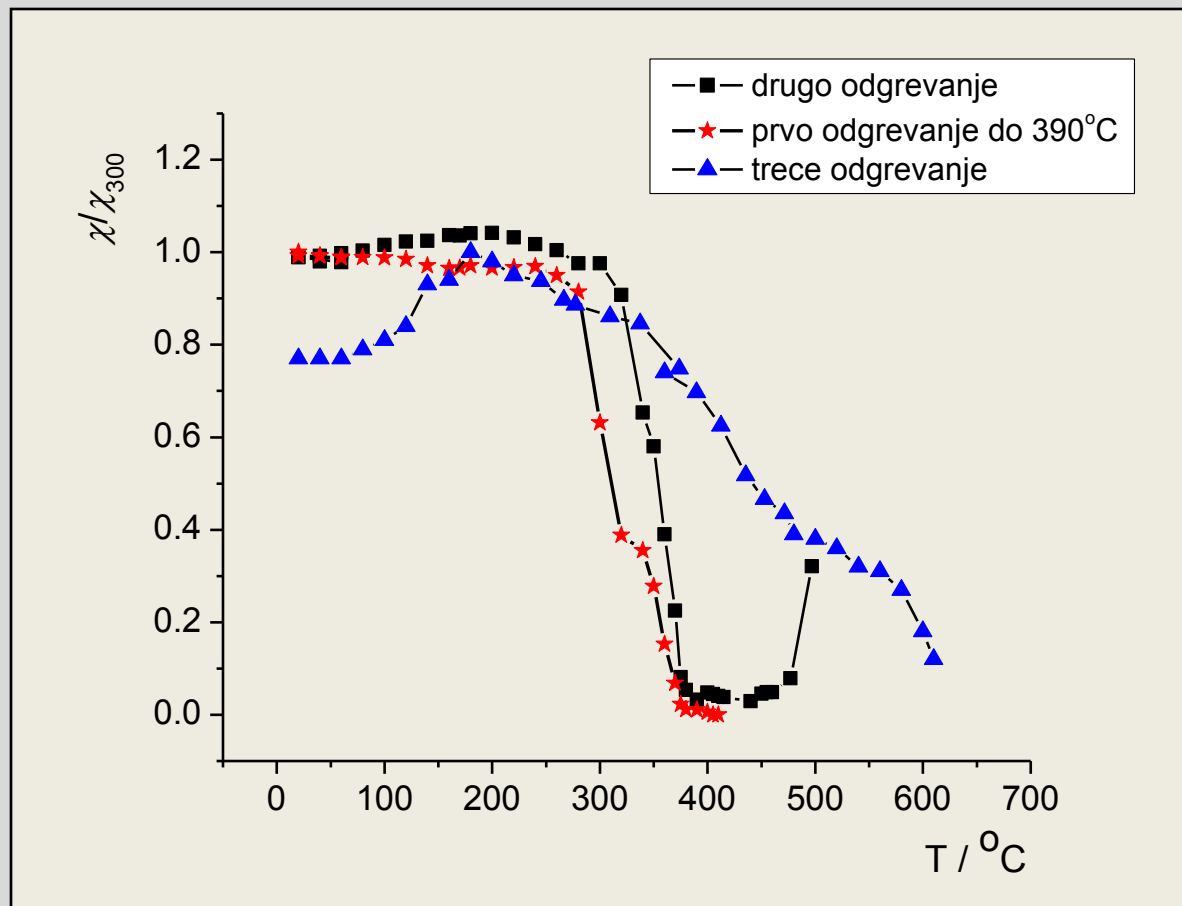
Promena električne otpornosti sa temperaturom za polaznu amorfnu leguru





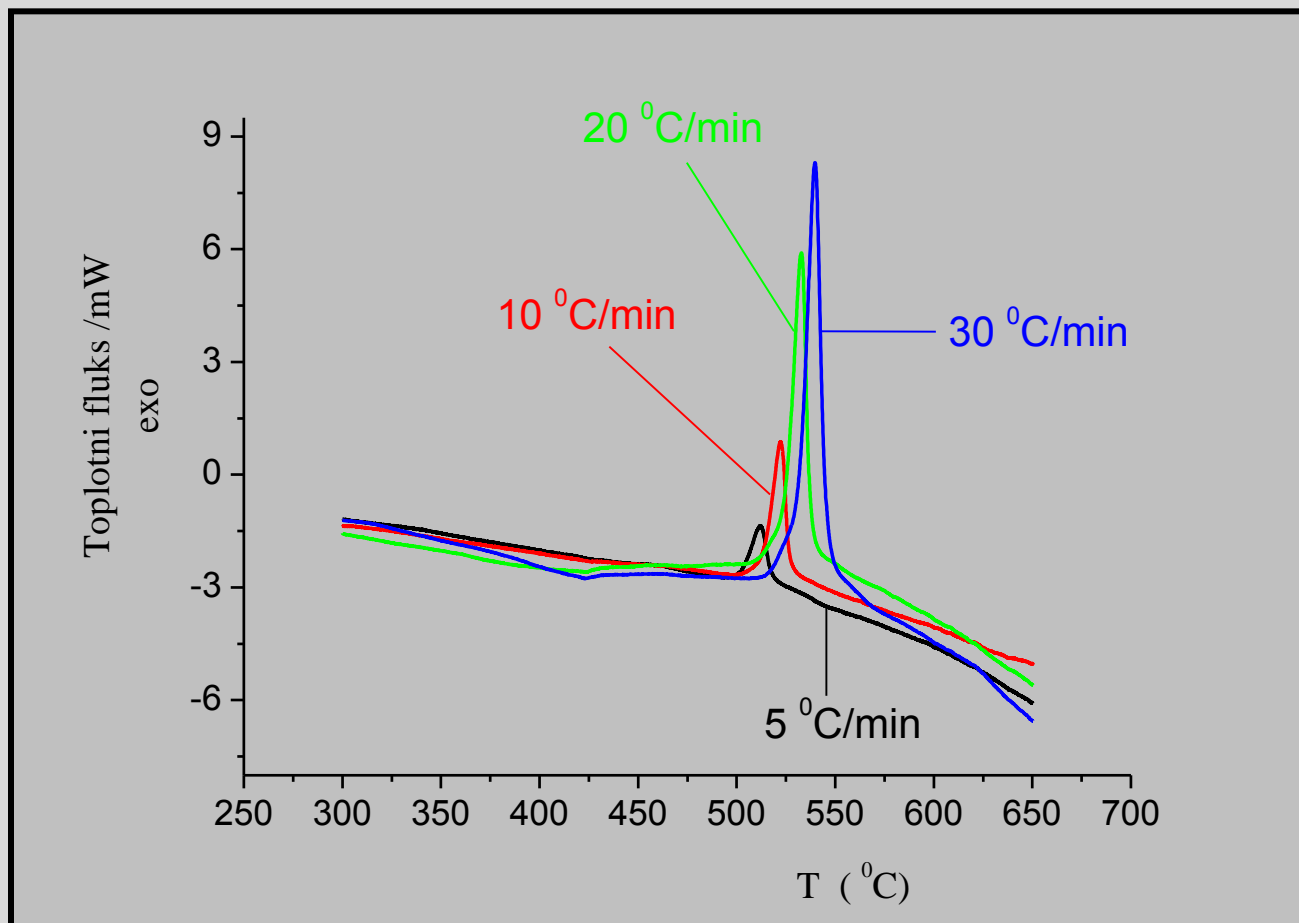
Promena električne otpornosti sa temperaturom za iskristalisanu leguru.

Uticaj strukturnih promena na magnetna svojstva



Temperaturska zavisnost relativne magnetne susceptibilnosti za tri ciklusa zagrevanja legure do različitih temperatura.

Kinetika kristalizacije

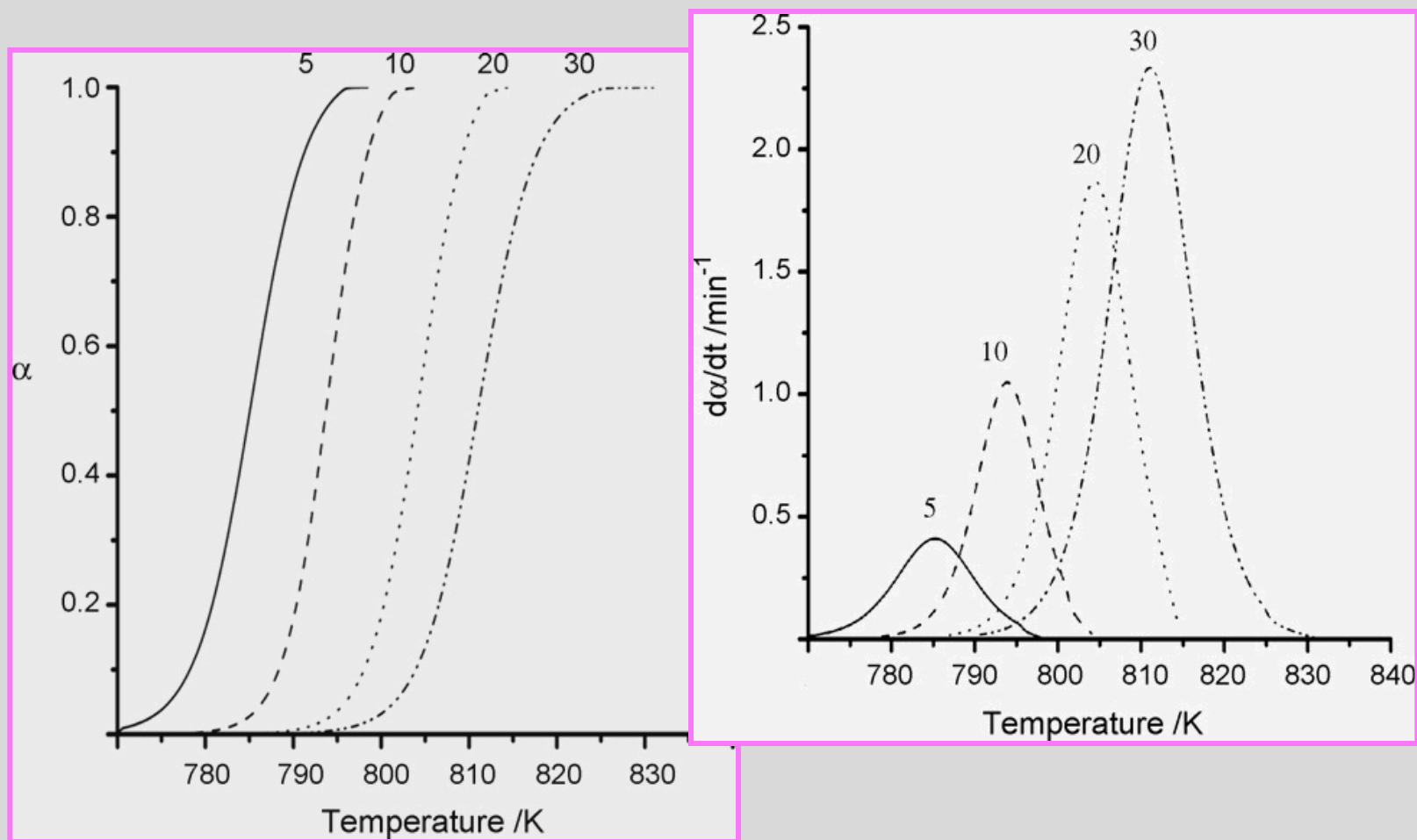


Egzo-kristalizaconi pikovi $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ amorfne legure za različite brzine zagrevanja.

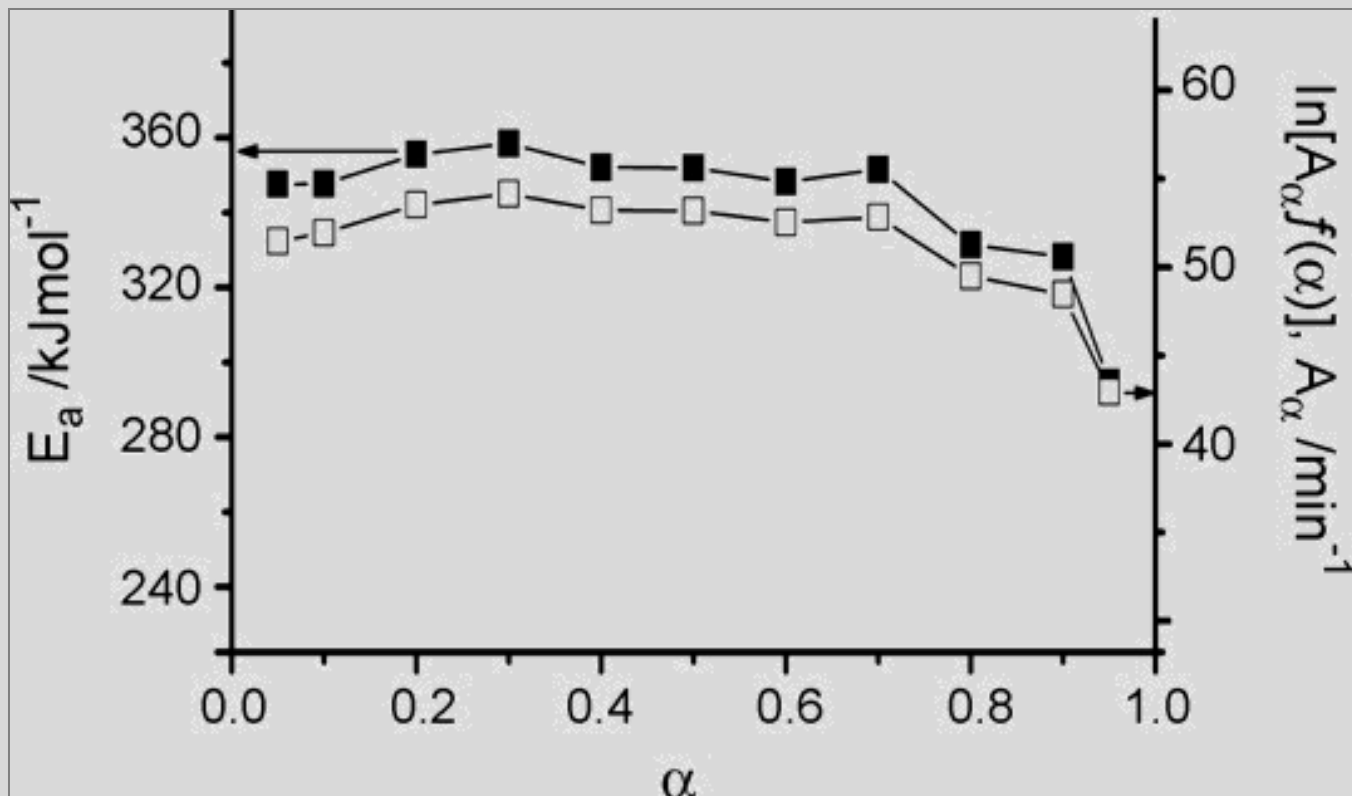
Kinetički parametri kristalizacije (prividna energija aktivacije, frekvencioni faktor, konstanta brzine) određeni po Ozawa-inoj metodi

β (K/min)	T_p (K)	E_a (kJ/mol)	A (min ⁻¹)	$k = Ae^{-(E_a/RT)}$ (min ⁻¹)
5	785,0	338,0±6	1,09±0.2 10 ²²	0,35
10	795,2			0,68
20	805,8			1,29
30	812,8			2,06

Stepen napredovanja reakcije

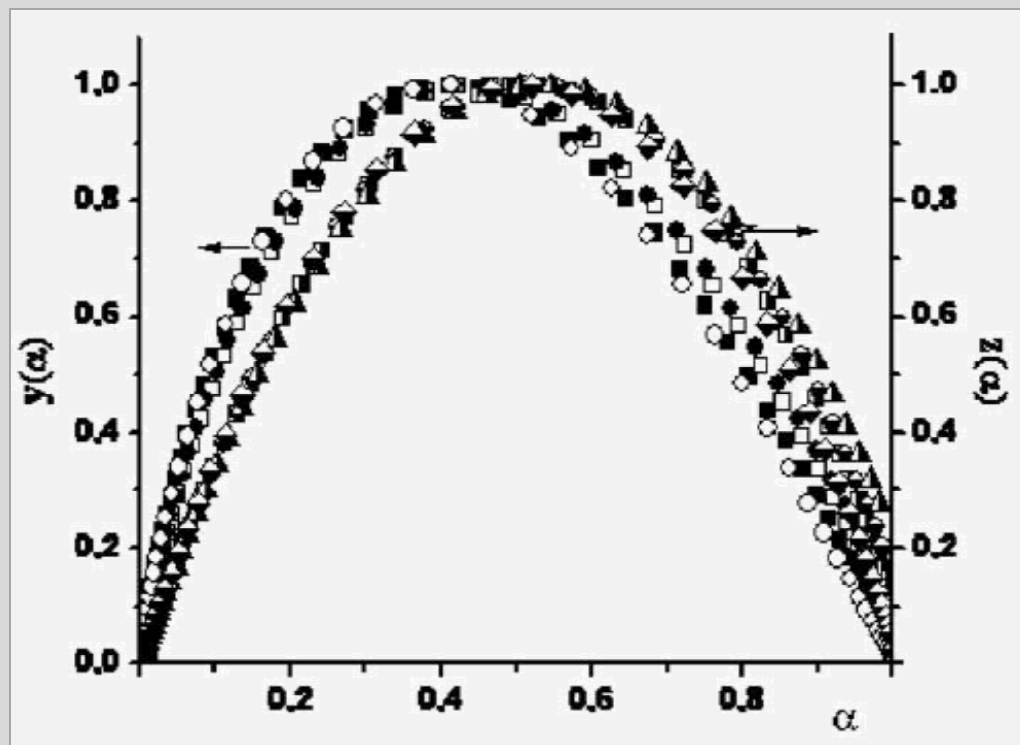


Promena kinetičkih parametara sa napredovanjem reakcije



E_a =konstantno za α veće od 0.7

Normalizovane Malekove funkcije za date brzine zagrevanja



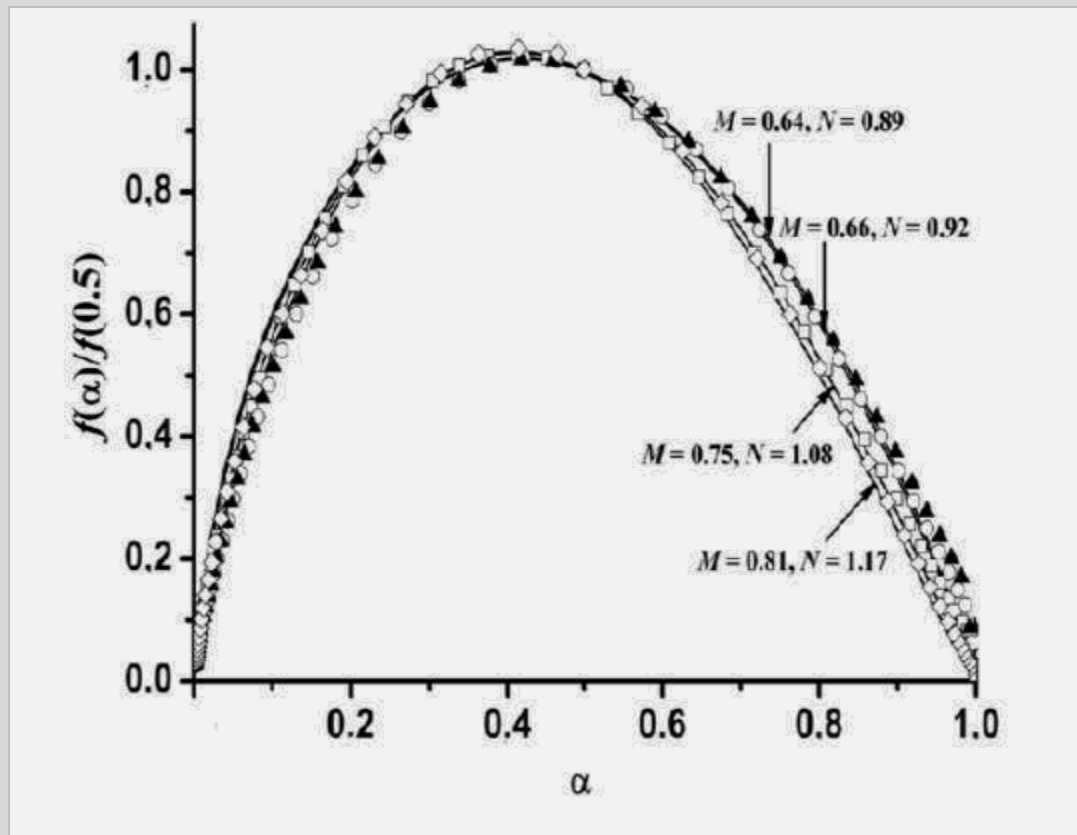
β K/min	$y(\alpha)^*$	$z(\alpha)^*$
5	0.41 ± 0.01	0.53 ± 0.01
10	0.42 ± 0.01	0.51 ± 0.01
20	0.42 ± 0.01	0.55 ± 0.03
30	0.41 ± 0.01	0.52 ± 0.01

Primena Šestak-Bergmanovog autokatalitičkog modela

$$f(\alpha) = \alpha^M (1 - \alpha)^N$$

β K/min	M	N	$\ln A$ 1/min
5	0.75 ± 0.03	1.08 ± 0.10	52.85 ± 0.06
10	0.66 ± 0.05	0.92 ± 0.05	53.03 ± 0.10
20	0.64 ± 0.05	0.89 ± 0.07	52.90 ± 0.07
30	0.81 ± 0.10	1.17 ± 0.04	53.02 ± 0.10
Srednja vrednost	0.72 ± 0.06	1.02 ± 0.07	52.95 ± 0.08

Primena Master-plot metode



$$\frac{d\alpha / dt}{(d\alpha / dt)_{\alpha=0.5}} \frac{\exp(E_a / RT)}{\exp(E_a / RT_{0.5})} = \frac{f(\alpha)}{f(0.5)}$$

Morfologija formiranih kristala

Matisuta-ina jednačina

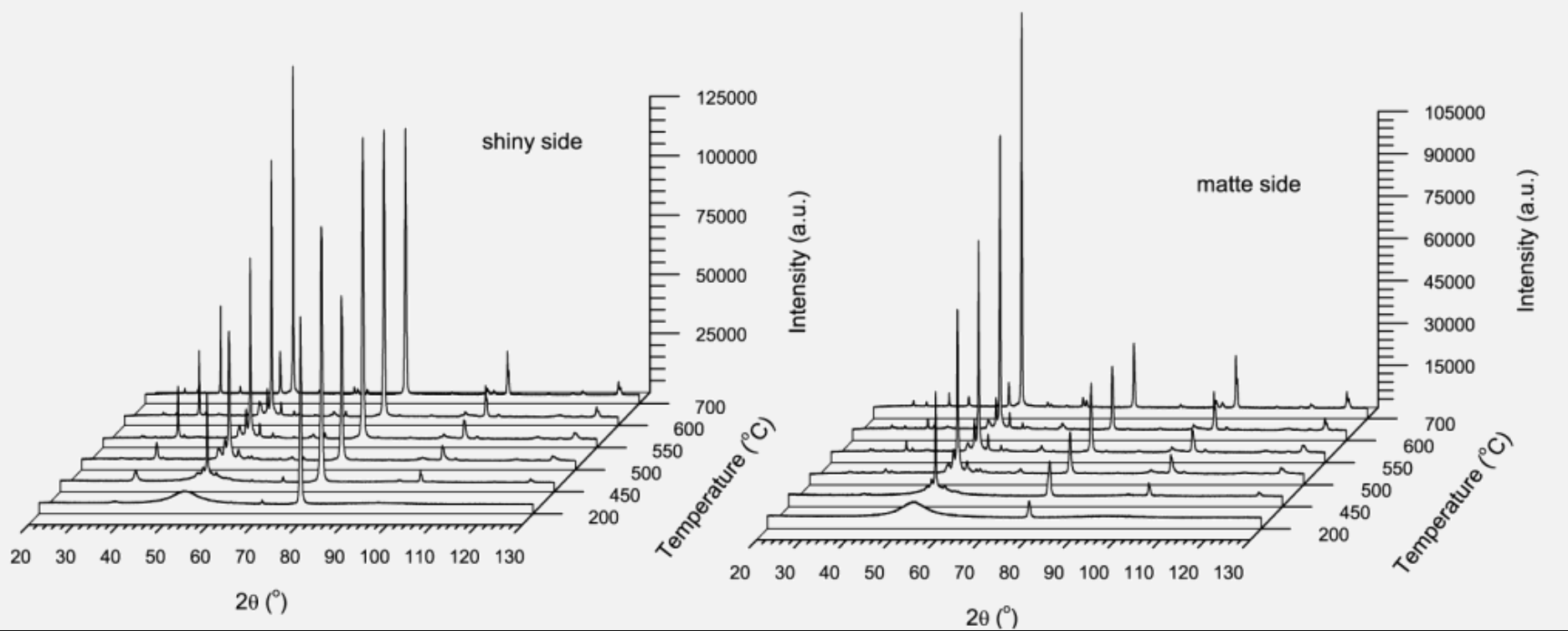
$$\ln[-\ln(1-\alpha)] = -n \ln \beta - \frac{1.052mE_a}{RT} + \text{const}$$

β K/min	m	s
n = 4		
5	2.84	1.16
10	3.08	0.92
20	3.22	0.78
30	2.84	1.16

Konstantna brzina
rasta kristala

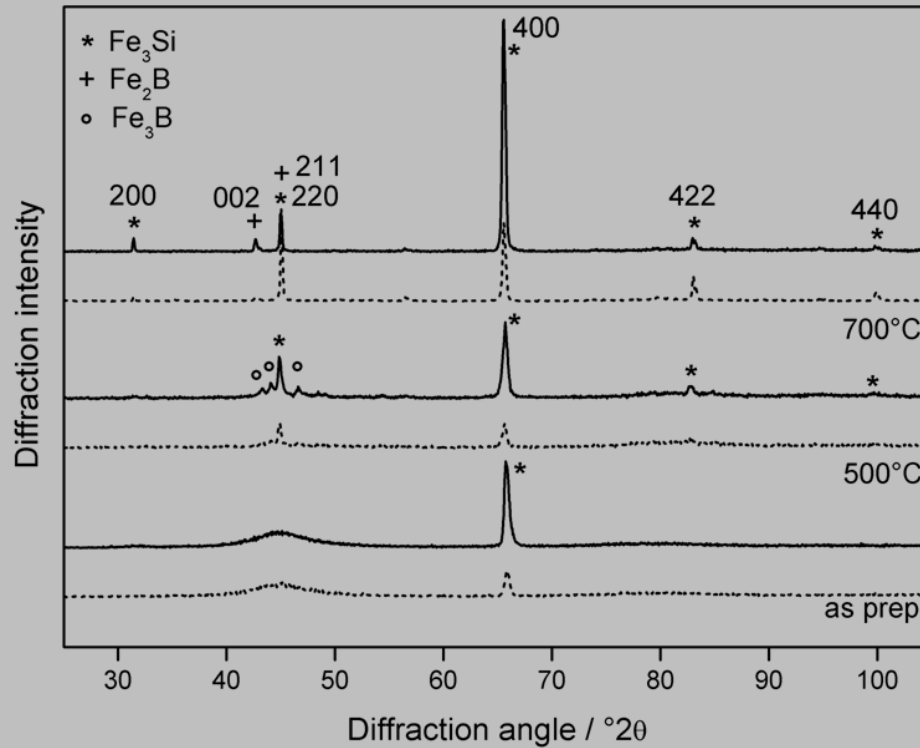
Trodimenzioni rast

Razlika morfologije kontaktne i slobodne strane trake



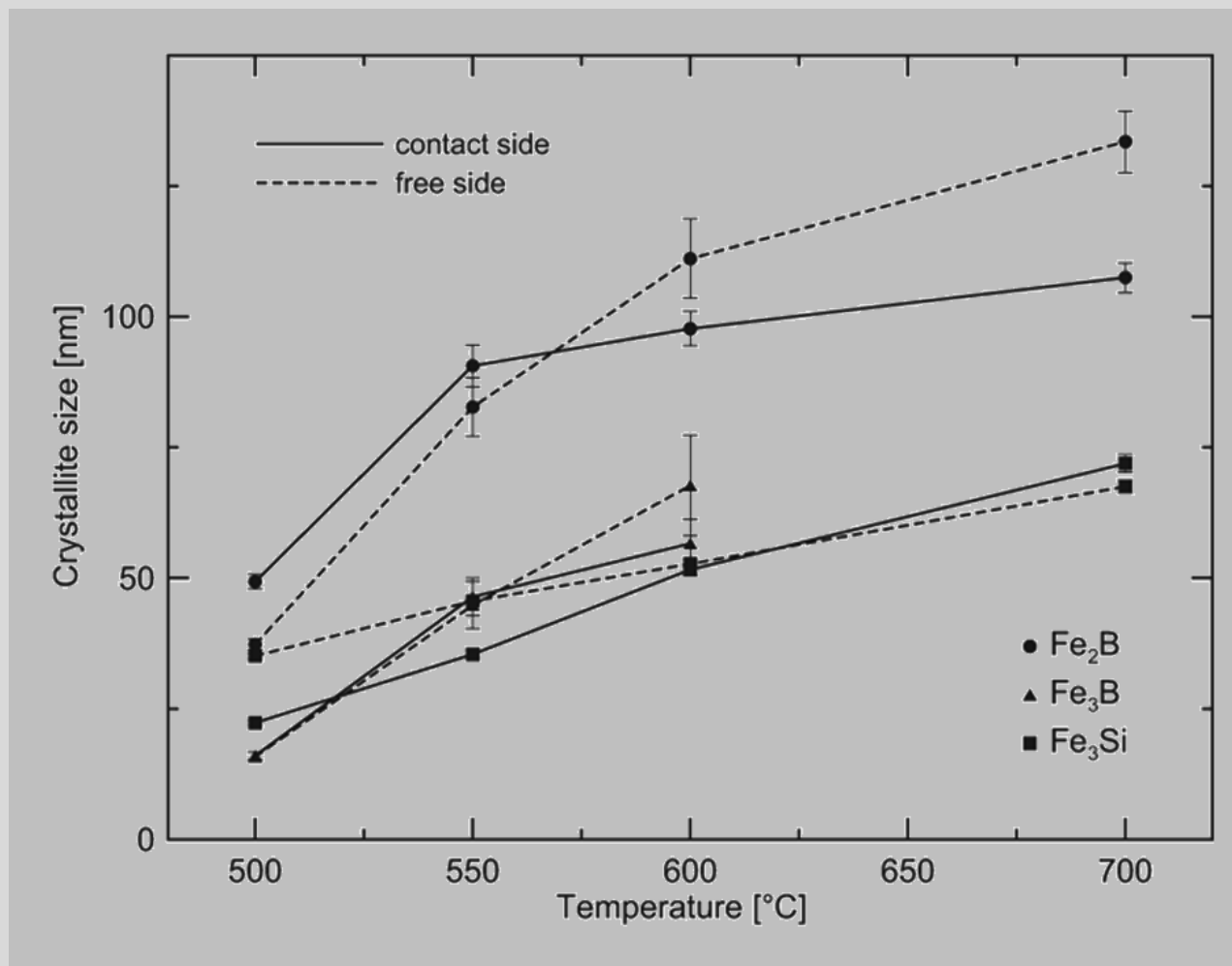
Kontaktna

Slobodna



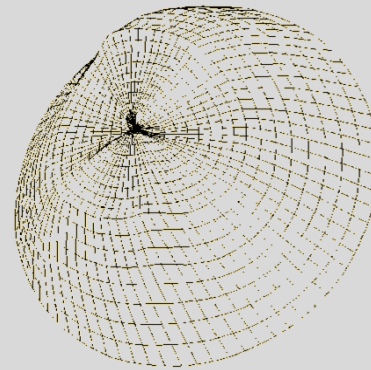
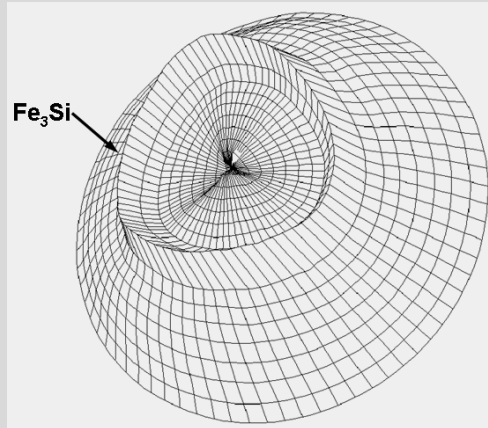
X-ray difraktogrami legure odgrevane na raznim temperaturama: difraktogrami slobodne strane dati su punim linijama a tačkicama difraktogrami kontaktne strane trake

Veličina kristalita na kontaktnoj i slobodnoj strani trake

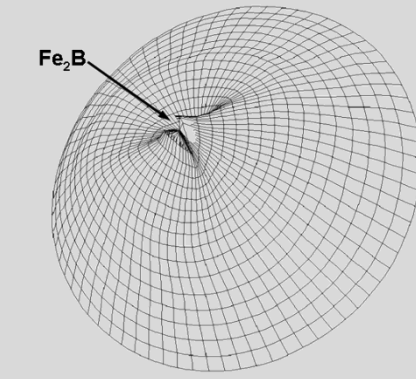
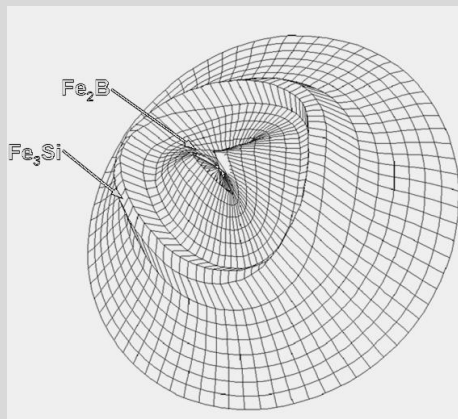


Tekstura

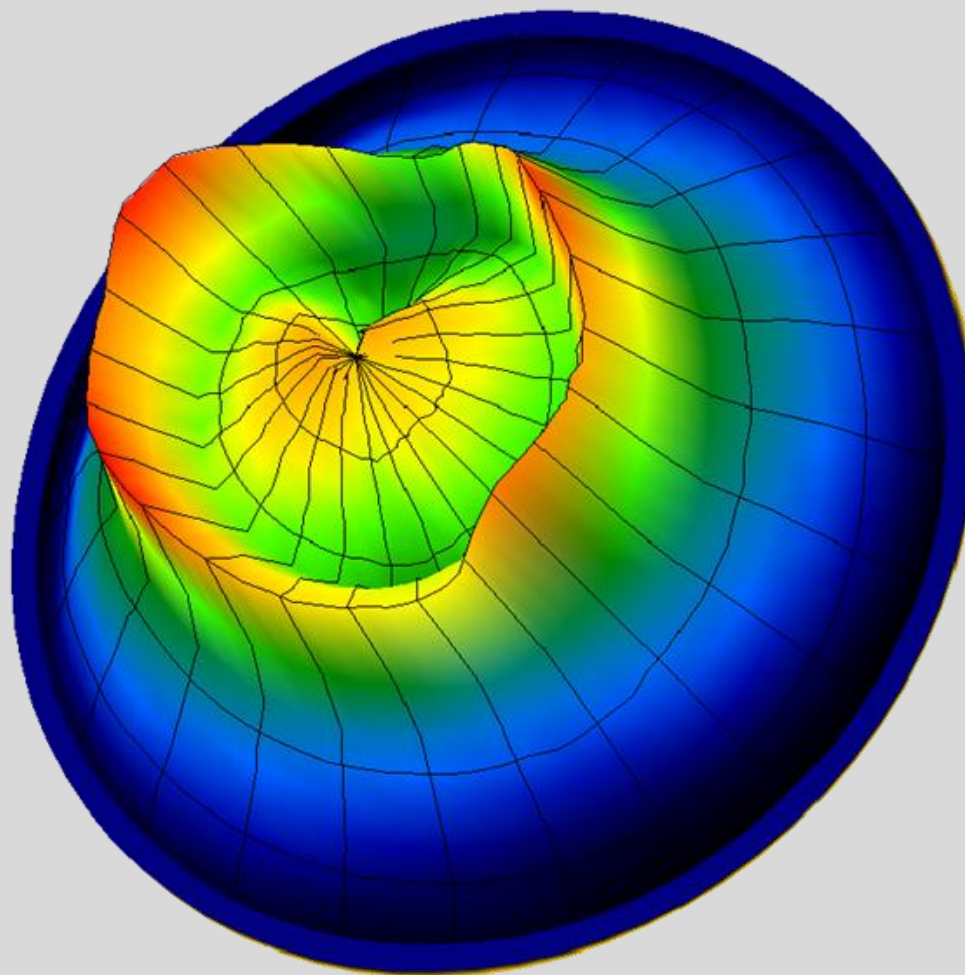
Pole-slike polazne legure slobodne i kontaktne trake na 45.0°



Pole-slike polazne legure slobodne strane odgrevane na 700 °C na 45.1 i 42.8 °



Legura odgrevvana na 730 C



ODF project:
S:_VANGERER-...PAT0224b.xrdml

Pole figure: 220 Raw

2Theta: 45.1000

Intensities:

	Psi	Phi	Intensity
Min	85.0	127.5	30.200
Max	45.0	212.5	2616.400

Dimension: 2.5D

Scale: Linear

Color map: X'Pert

Contours: 10

	Intensity	Color
1	265.309	
3	735.527	
5	1205.745	
7	1675.964	
10	2381.291	

Grid settings:

	Psi	Phi
First	0	0
Last	90	360
Step	15	15

Literatura

D. M. Minić, B. Adnadjević, *Mechanism and Kinetics of Crystallization of α -Fe in Amorphous $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ Alloy*, Thermochim. Acta 424(2008) 41-46.

D. M. Minić, D. G. Minić and A. Maričić, *Stability and Crystallization of $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ Amorphous Alloy*, J. Non-Crystalline Solids 355(2009) 2503-2507.

D. M. Minić, A. Maricić, B. Adnadjević, *Crystallization of α -Fe Phase in Amorphous $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ Alloy*, Journal of Alloys and Compounds 473(2009) 363-367.

Borivoj Adnađević, Bojan Janković, Dragica M. Minić, *Kinetics of the apparent isothermal and non-isothermal crystallization of the α -Fe phase within the amorphous $\text{Fe}_{81}\text{B}_{13}\text{Si}_4\text{C}_2$ alloy*, J. Physic.Chem.Solids, 71(2010) 927-934

D. M. Minić, M. Plazinić, Živanić, A. Maričić, *Influence of structural transformations on electric and magnetic properties of $Fe_{81}B_{13}Si_4C_2$ amorphous alloy*, Science of Sintering 42(1)(2010)61-68.

D. M. Minić, D. M. Minić, T. Žák, P. Roupcová, B. David, *Structural Transformations of $Fe_{81}B_{13}Si_4C_2$ Amorphous Alloy Induced by Heating*, J. Magn. Magn. Mater. 323 (2011) 400-404.

Dragica M. Minić, Vladimir A. Blagojević, Dušan M. Minić, Tomáš Žák, *Influence of microstructural inhomogeneity of individual sides of $Fe_{81}Si_4B_{13}C_2$ amorphous alloy ribbon on thermally induced structural transformations*, Mater.Phys.Chem 130(2011) 980-985.

Dragica.M. Minić, Vladimir. A. Blagojević, Dušan.M. Minić, Aleksandra. Gavrilović, Lidija Rafailović, Tomas. Žak, *Influence of microstructure on microhardness of $Fe_{81}Si_4B_{13}C_2$ amorphous alloy after thermal treatment*, Metallurgical Materials Transactions A, 42A(2011)4106-4112.

• D.M. Minić, V. A. Blagojević, D.M. Minić, T. Žak, *Influence of microstructural inhomogeneity of individual sides of $Fe_{81}Si_4B_{13}C_2$ amorphous alloy ribbon on thermally induced structural transformations*, Mater.Phys. Chem, 130(2011) 980-985.

Dušan M. Minić, Vladimir A . Blagojević, Dragica M. Minić, *Uticaj zagrevanja na funkcionalna svojstva amorfne legure $Fe_{81}B_{13}Si_4C_2$* , Tehnika 3 (2013)239-250.

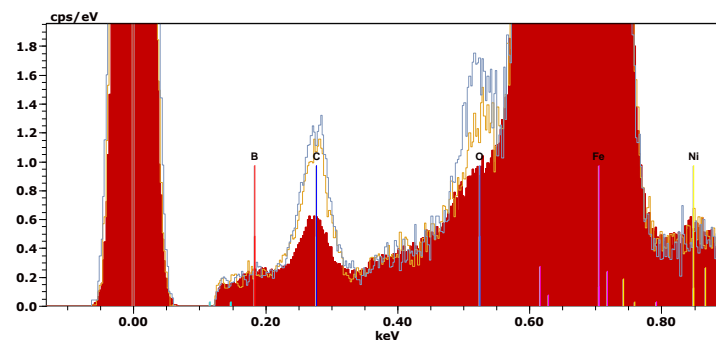
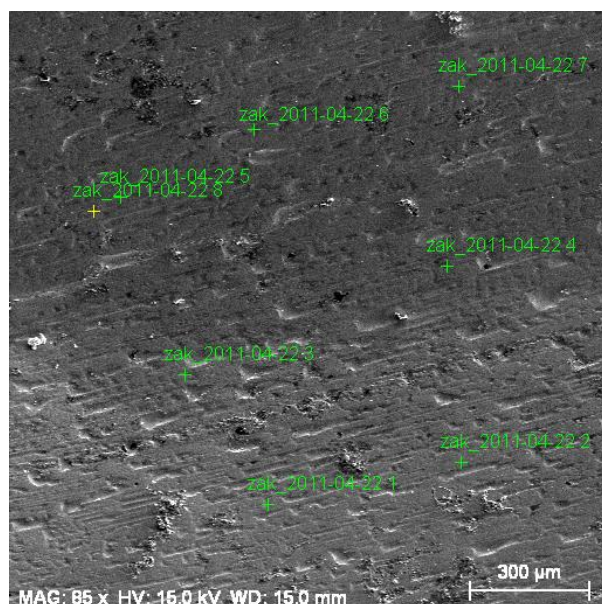
Milica Vasić, Dušan Minić, Vladimir Blagojević, Dragica Minić, *Mechanism and kinetics of crystallization of amorphous $Fe_{81}B_{13}Si_4C_2$ alloy*, Thermochemica Acta, 572(2013) 45-50.

Amorfna $\text{Fe}_{75}\text{Ni}_2\text{Si}_8\text{B}_{13}\text{C}_2$ legura

Hemijski sastav

Polazna legura

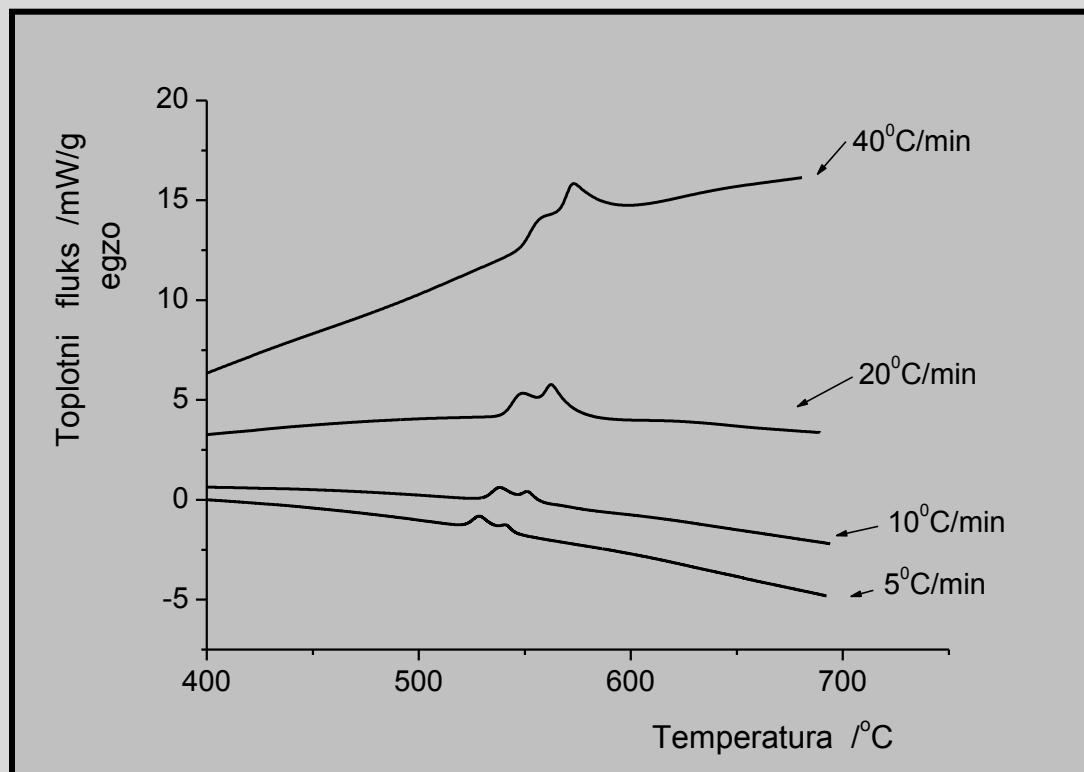
Surface of the sheet



Mass percent (%)

Spectrum	B	C	O	Al	Si	Fe	Ni
zak_2011-04-22 1	1.21	5.60	0.70	0.45	4.79	85.25	2.00
zak_2011-04-22 2	2.80	1.86	0.67	0.00	4.39	88.76	1.51
zak_2011-04-22 3	2.64	2.67	1.18	0.03	4.37	87.22	1.88
zak_2011-04-22 4	3.73	3.37	1.29	0.06	4.45	85.72	1.39
zak_2011-04-22 5	7.16	4.48	0.89	0.06	4.26	81.76	1.39
zak_2011-04-22 6	4.89	5.16	2.88	0.05	4.14	81.48	1.38
zak_2011-04-22 7	5.64	2.06	1.05	0.02	4.48	84.88	1.87
zak_2011-04-22 8	0.00	1.51	0.80	0.05	4.63	91.34	1.66
Mean value:	3.51	3.34	1.18	0.09	4.44	85.80	1.64
Sigma:	2.35	1.58	0.72	0.15	0.20	3.33	0.25
Sigma mean:	0.83	0.56	0.26	0.05	0.07	1.18	0.09

Termička stabilnost

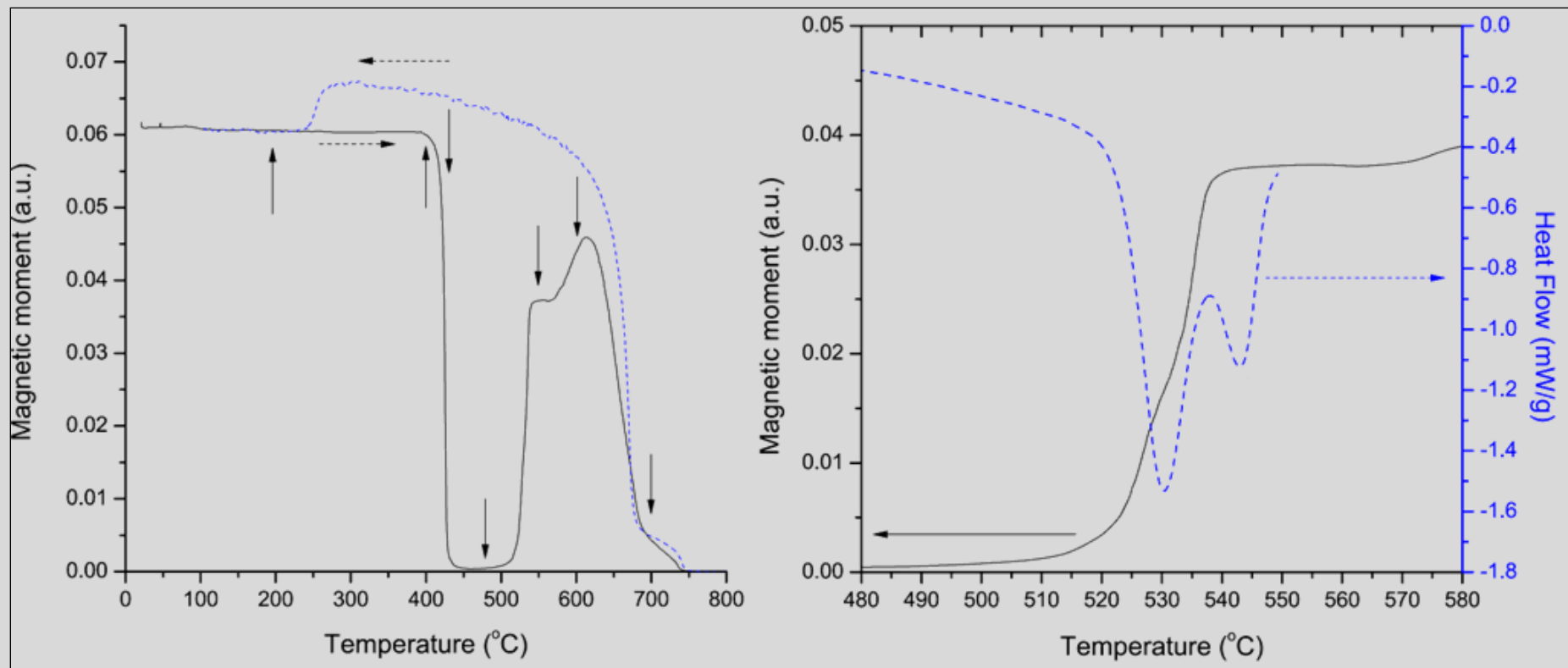


DSC krive legure u protočnoj struji azota dobijene pri različitim brzinama zagrevanja.

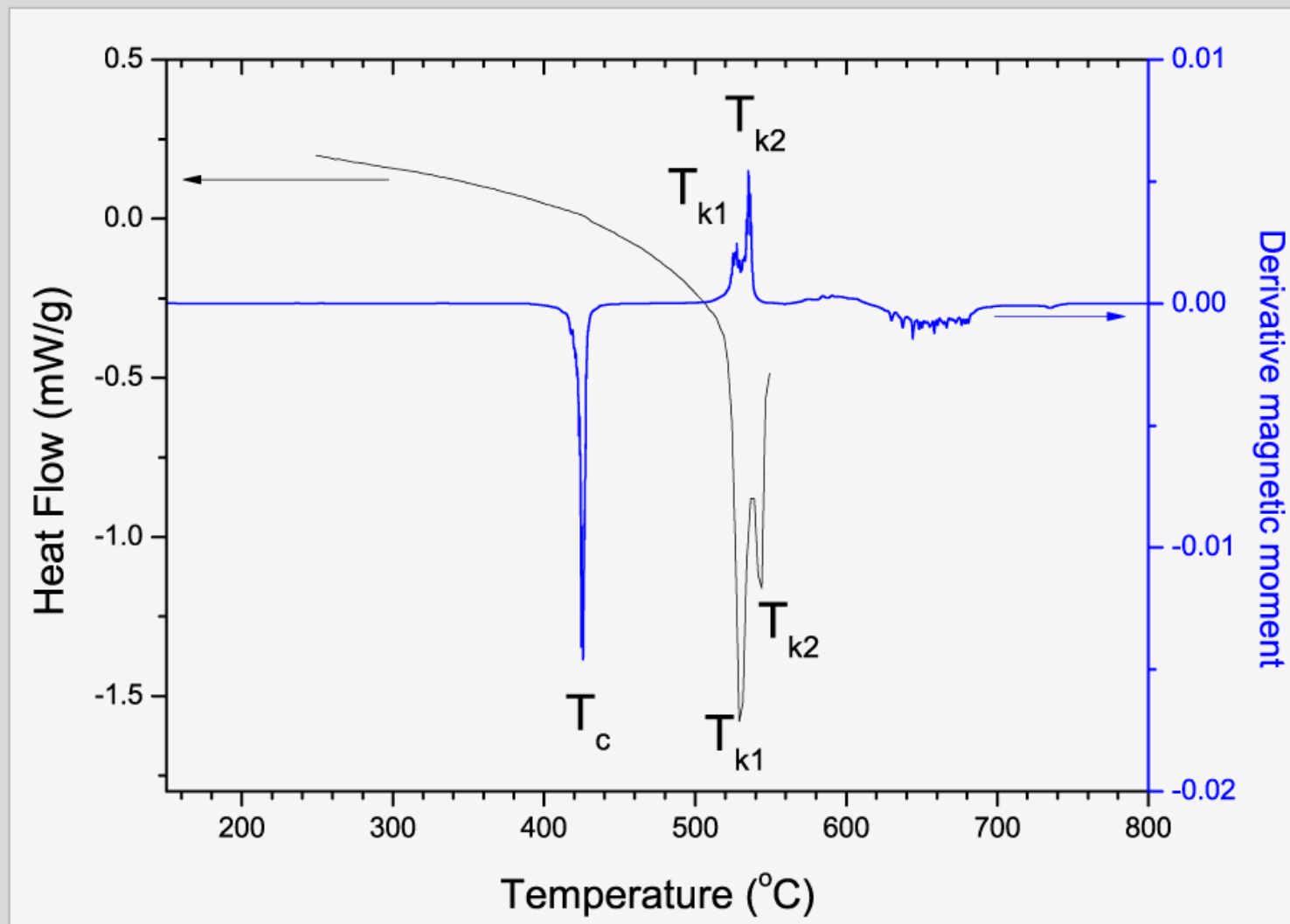
Kinetički parametri oba kristalizaciona stupnja određena po metodama Kissinger-a i Ozawa-e.

	Prvi stupanj			Drugi stupanj		
β	T_p^1	E_a^1	$\ln A^1$	T_p^2	E_a^2	$\ln A^2$
K/min	K	kJ/mol		K	kJ/mol	
5	801,7	380,0±8 (Kissinger)	55,96 (Kissinger)	814,5	350,0±8 (Kissinger)	51,76 (Kissinger)
10	811,4	394,0±8 (Ozawa)	71,38 (Ozawa)	824,3	370,0±8 (Ozawa)	51,35 (Ozawa)
20	822,0			835,6		
40	830,5			846,1		

Thermomagnetna i DSC kriva



DSC kriva (5 oC/min) i derivativna kriva magnetnog momenta (4 oC/min)



Strukturne transformacije izazvane zagrevanjem

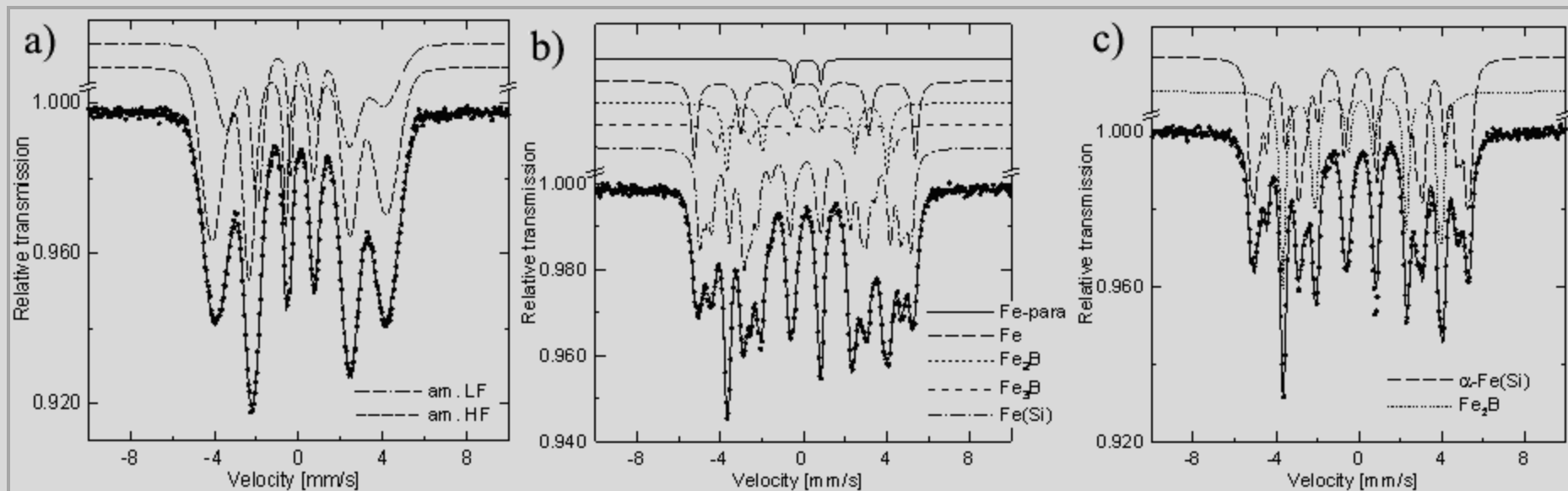
Mössbauer spectri

LF i HF označavaju komponentu niskog i visokog polja:

(a) Polazna legura

(b) Legura grejana na 550°C

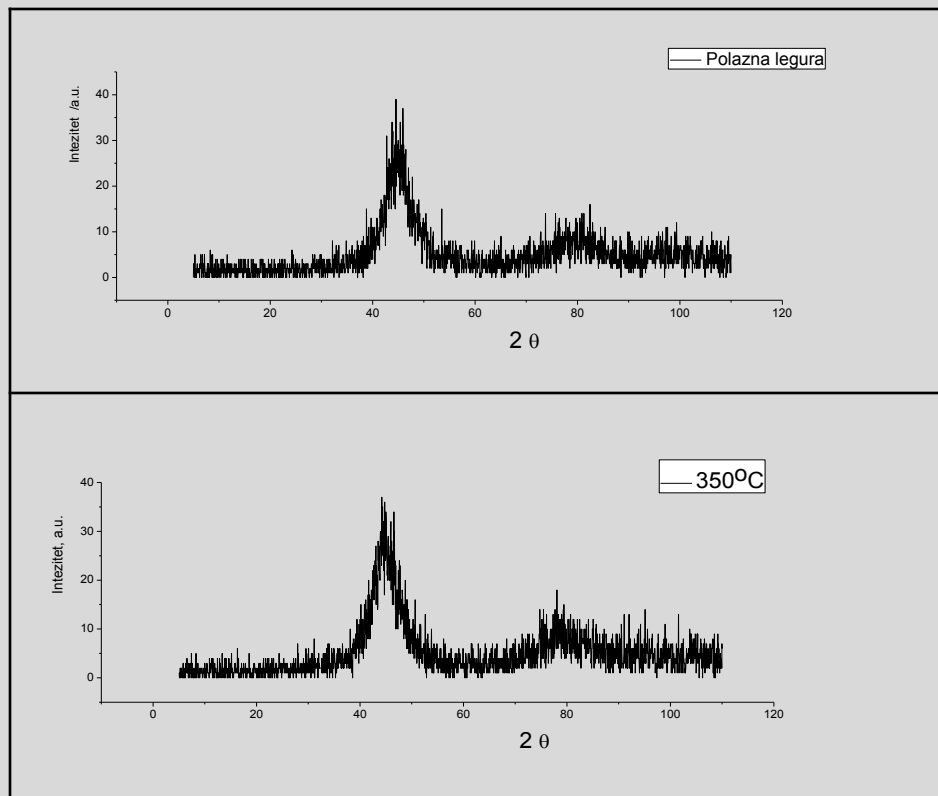
(c) Legura grejana na 700°C.



Mössbauer-ova tentativna fazna analiza
(distribucija Fe atoma)

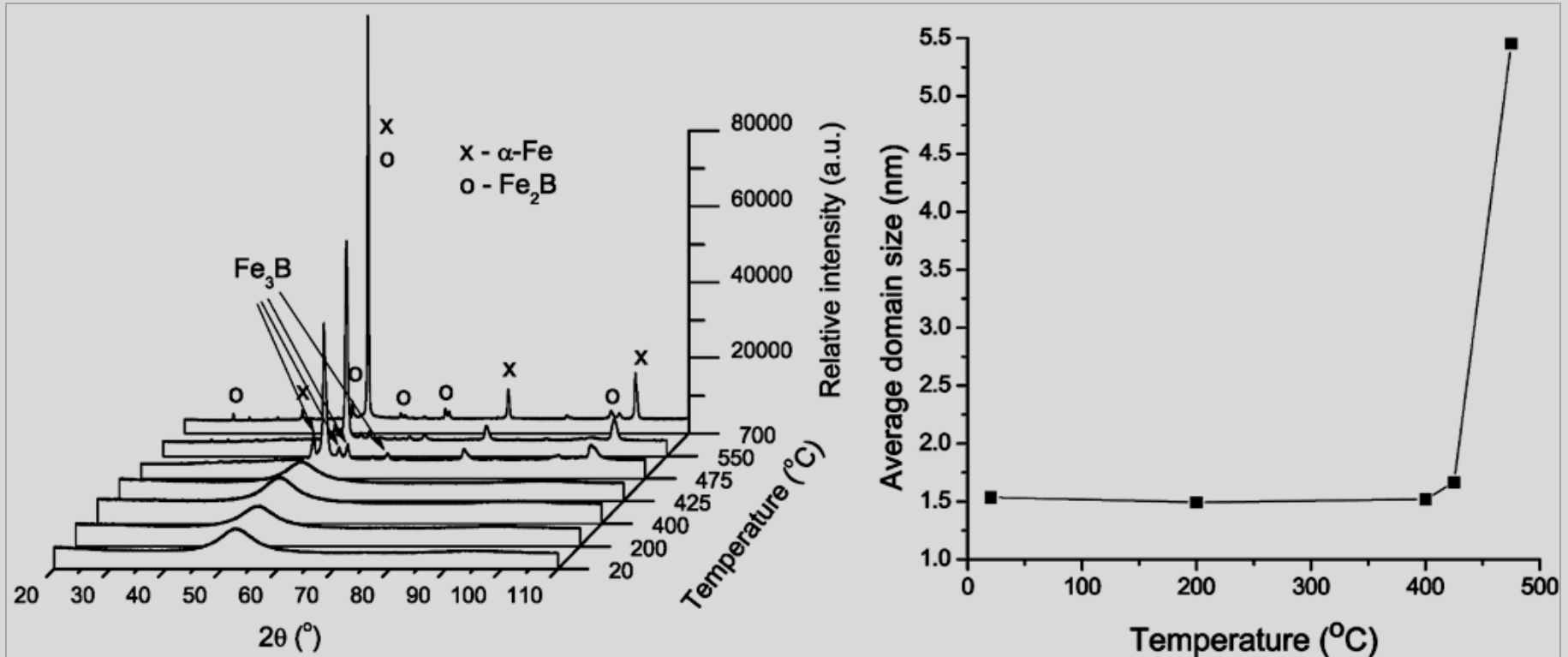
Temperatura	Amorfno (at% Fe)	α -Fe(Si) (at% Fe)	α -Fe (at% Fe)	FeB (at% Fe)	Fe ₃ B (at% Fe)	Fe ₂ B (at% Fe)	Fe para (at% Fe)
25°C	1,00	-	-	-	-	-	-
200 °C	1,00	-	-	-	-	-	-
400 °C	1,00	-	-	-	-	-	-
425 °C	0,92	0,06	-	0,2	-	-	-
475 °C	0,66	0,08	0,05	-	0,21	-	-
550 °C	-	0,57	0,07	-	0,13	0,22	0,01
600 °C	-	0,64	-	-	-	0,36	-
700 °C	-	0,64	-	-	-	0,36	-

Struktura polazne legure



Difraktogrami X-zraka polazne legure i
uzorka legure odgrejanog na 350°C

XRD spectra i promena veličine domena
uređenih na blizinu sa temperaturom zagrevanja
legure

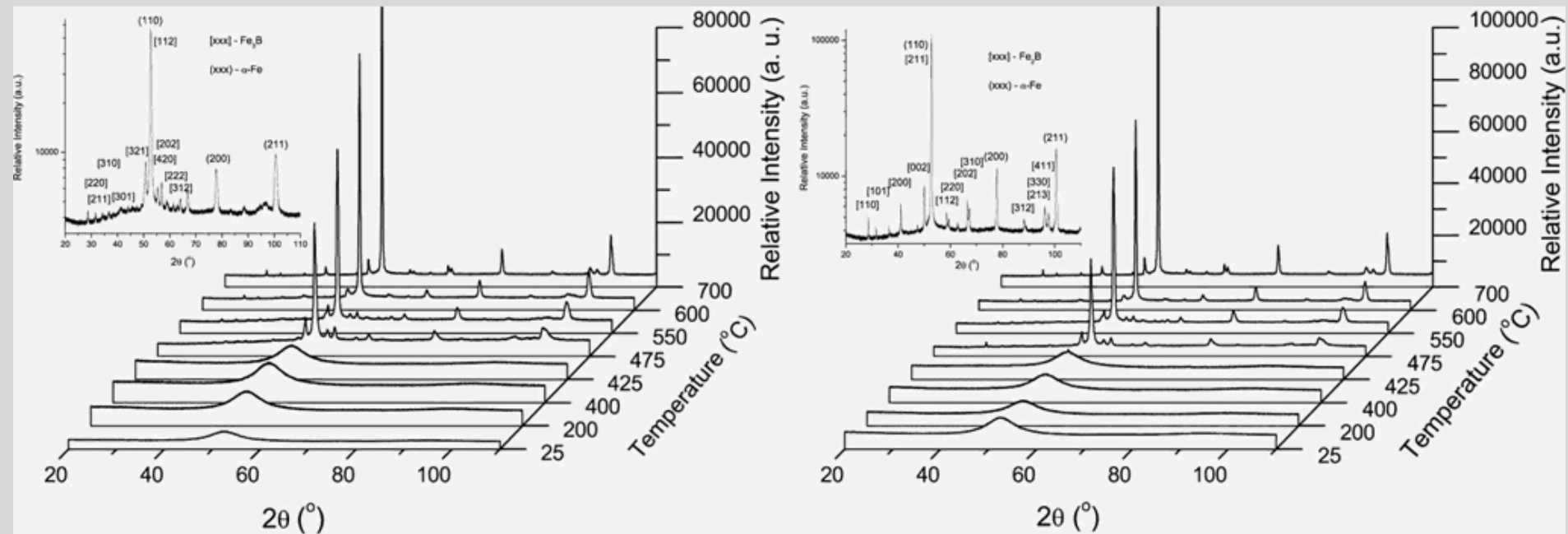


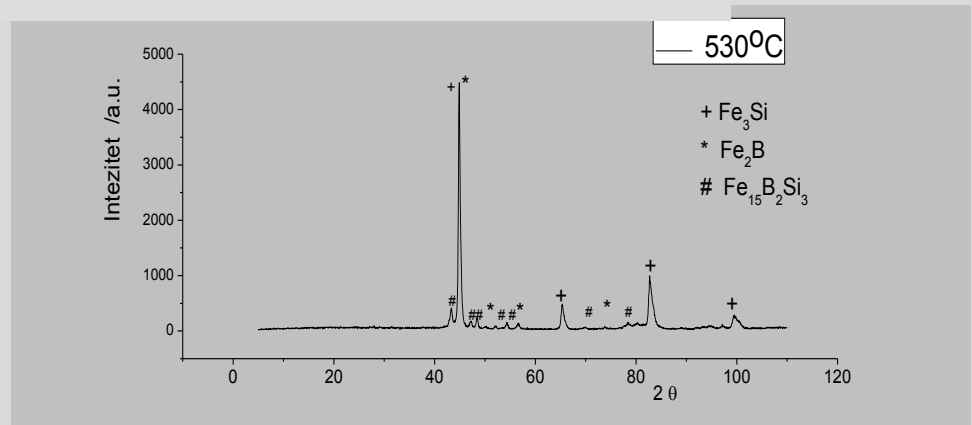
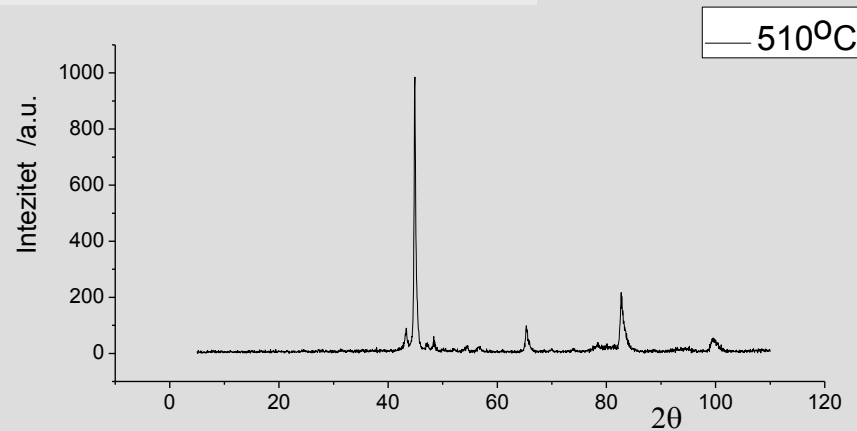
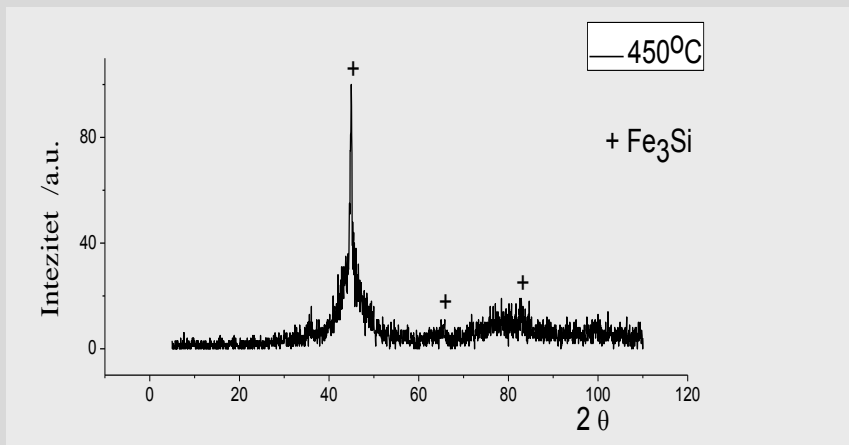
Strukturne transformacije

X-ray difraktogrami kontaktne i slobodne površine

isečak (levo): uzorak odgrejan na 475oC, sa pikovima α -Fe i Fe₃B faza;

Isečak (desno) : uzorak odgrejan na 700oC, sa pikovima α -Fe i Fe₂B faza.





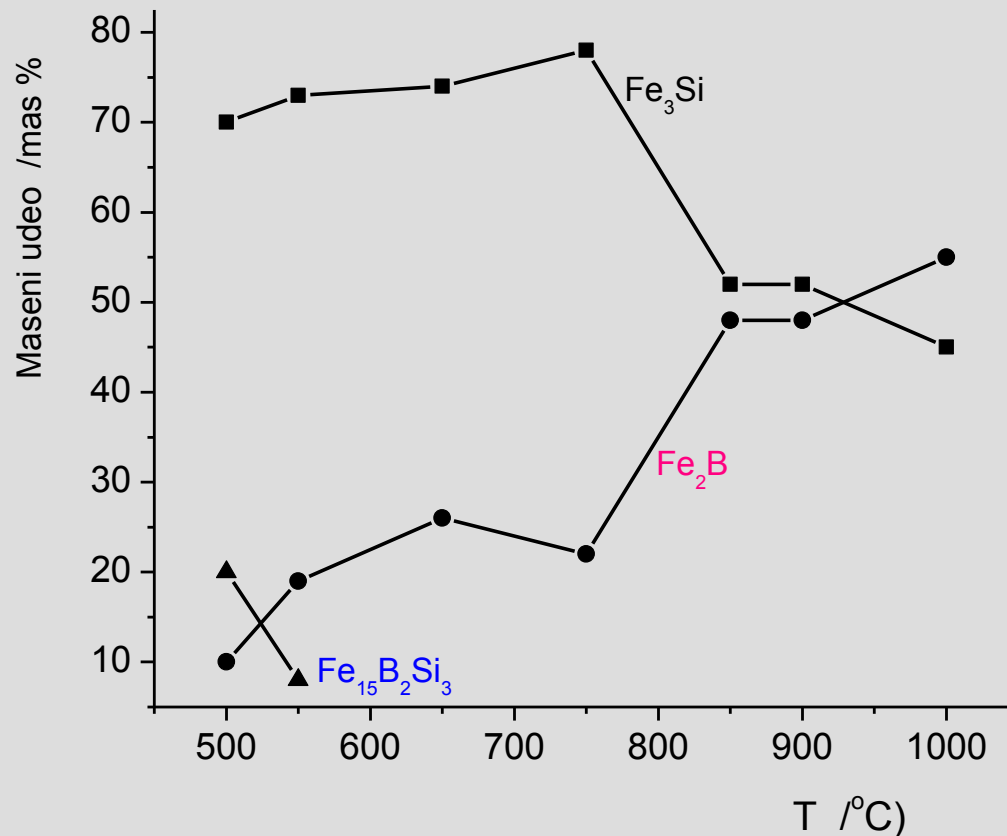
Difraktogrami X-zraka
uzoraka legure odgrevane
na raznim temperaturama.

Kristalna simetrija, prostorna grupa (Hermann-Mauguin) i parametri rešetke nađenih faza u iskristalisanjoj leguri

Faza	Prostorna grupa	Kristalni sistem	Parametri rešetke [Å]	
			a	c
Fe ₂ B [ICDD-PDF 00-036-1332]	I4/ncm	Tetragonalni	5.110	4.249
Fe ₃ Si [ICDD-PDF 00-042-1329]	Fm-3m	Kubni	5.662	-
Fe ₃ B [ICDD-PDF 00-039-1316]	I-4	Tetragonalni	8.674	4.313

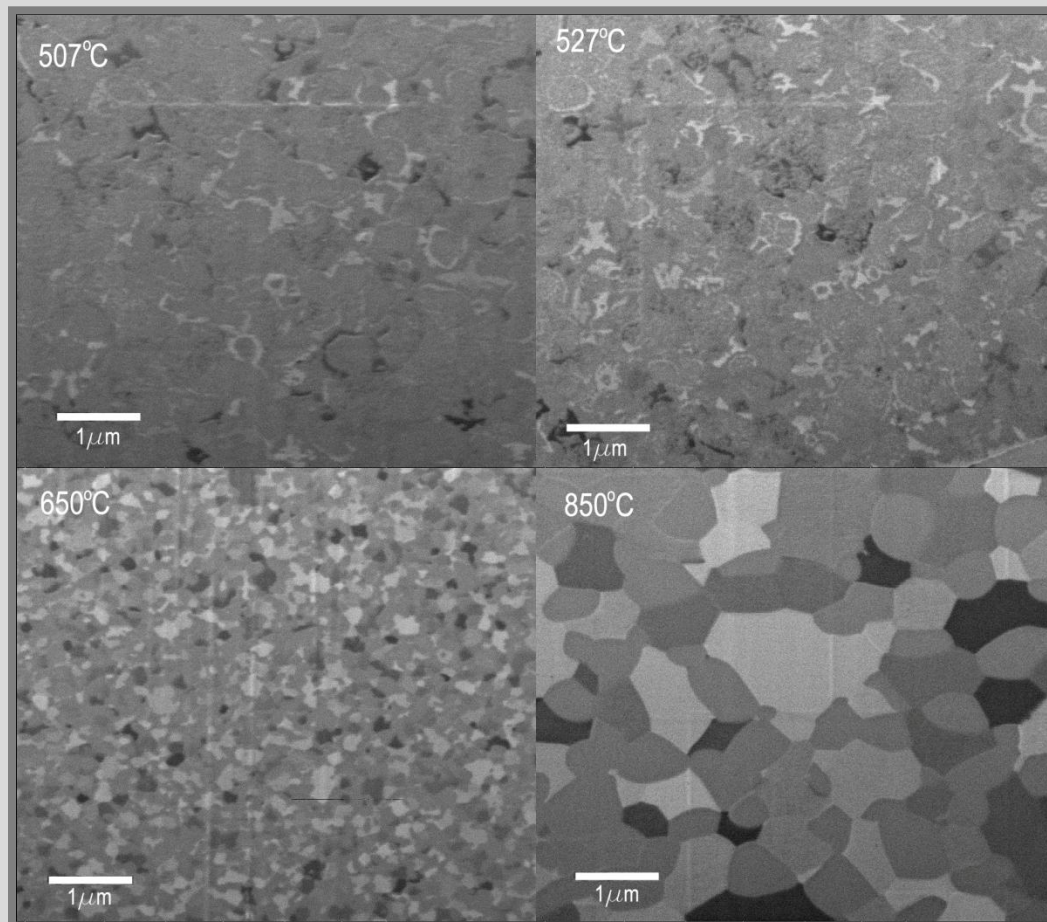
XRD fazna analiza (fazni sadržaj u mas%,
prosečna veličina kristala u nm)

Temperatura odgrevanja	Amorfno	α -Fe(Si) #103624		Fe3B		Fe2B #391328	
	mas%	mas%	d[nm]	mas%	d[nm]	mas%	d[nm]
polazna legura	100	-	-	-	-	-	-
200 °C	100	-	-	-	-	-	-
400 °C	100	-	-	-	-	-	-
425°C	100	-	-	-	-	-	-
475°C	34	48	18	18	15	-	-
550 °C	-	71	27	15	28	14	19
600 °C	-	76	49	-	-	24	22
700 °C	-	71	83	-	-	23	58

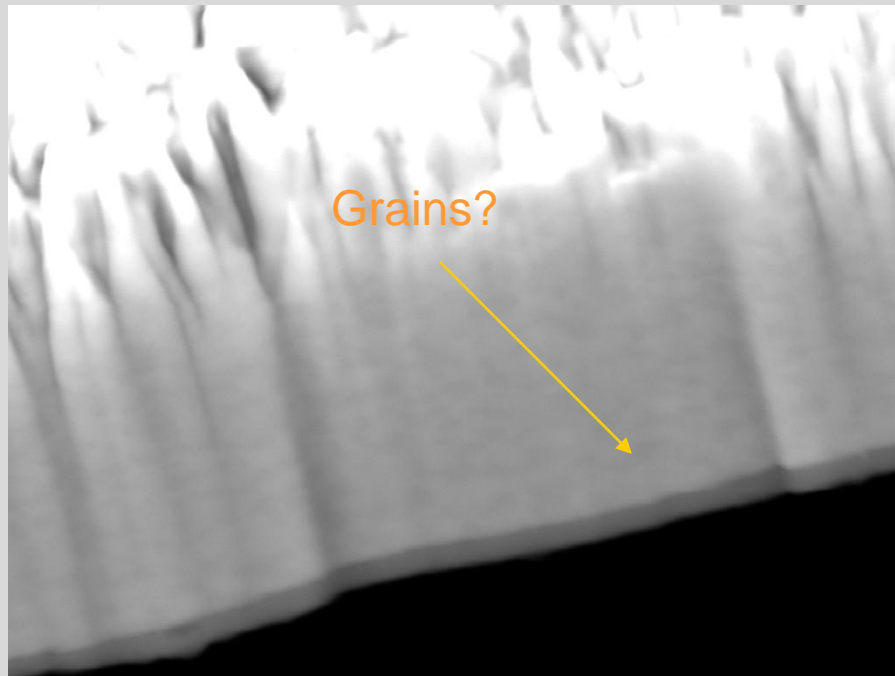


Maseni udeli nađenih kristalnih faza određeni metodom Rietveld-ovog utančavanja u funkciji temperature odgrevanja uzorka.

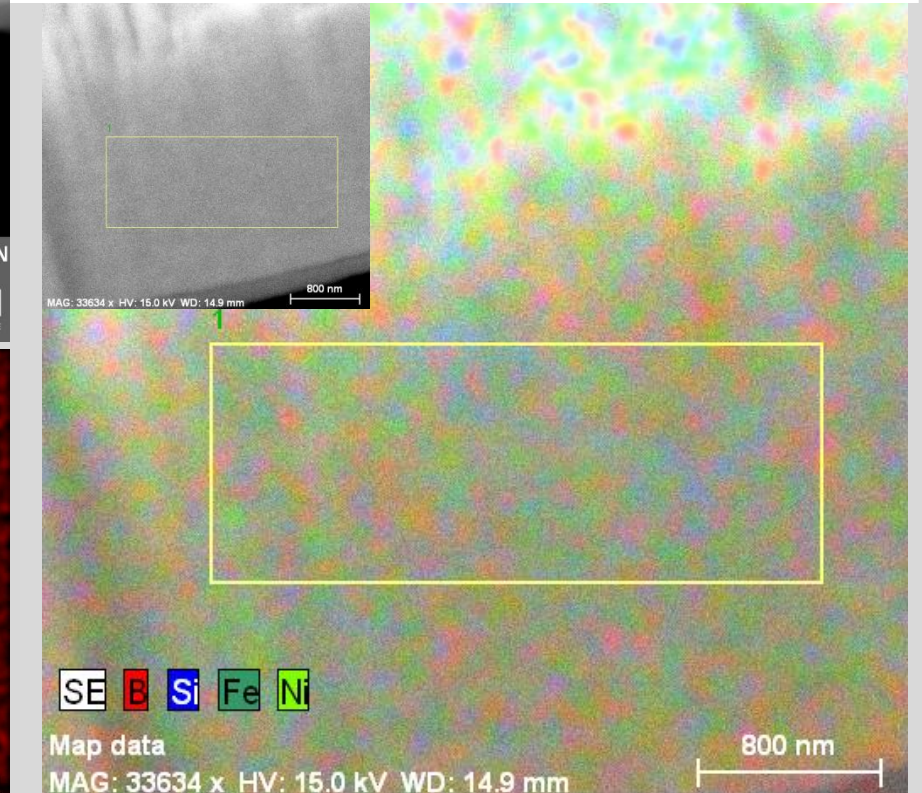
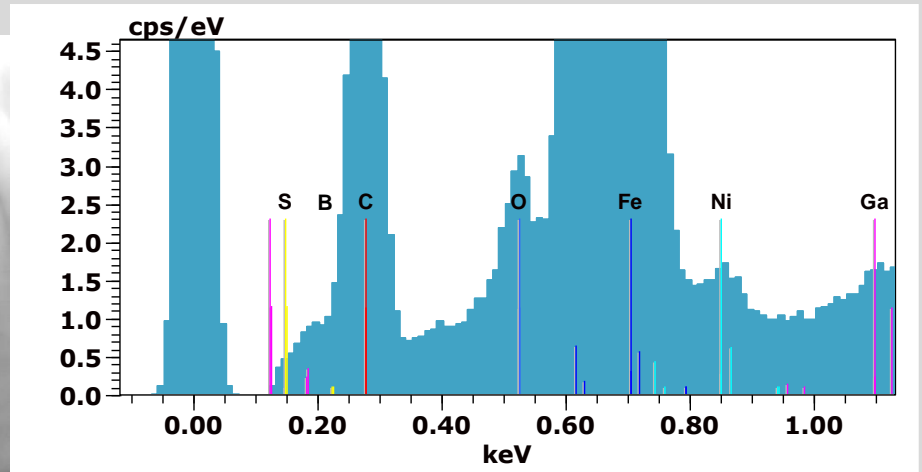
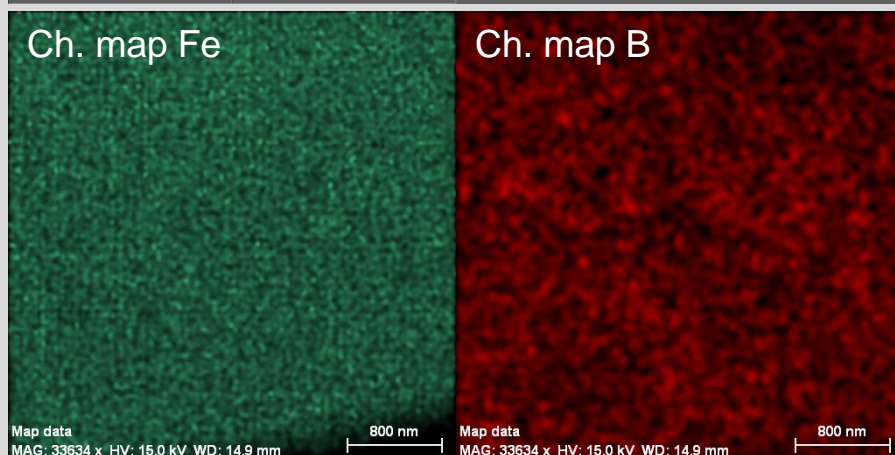
- FIB-SEM mikrografije legure $\text{Fe}_{75}\text{Ni}_2\text{Si}_8\text{B}_{13}\text{C}_2$ posle odgrevanja na raznim temperaturama



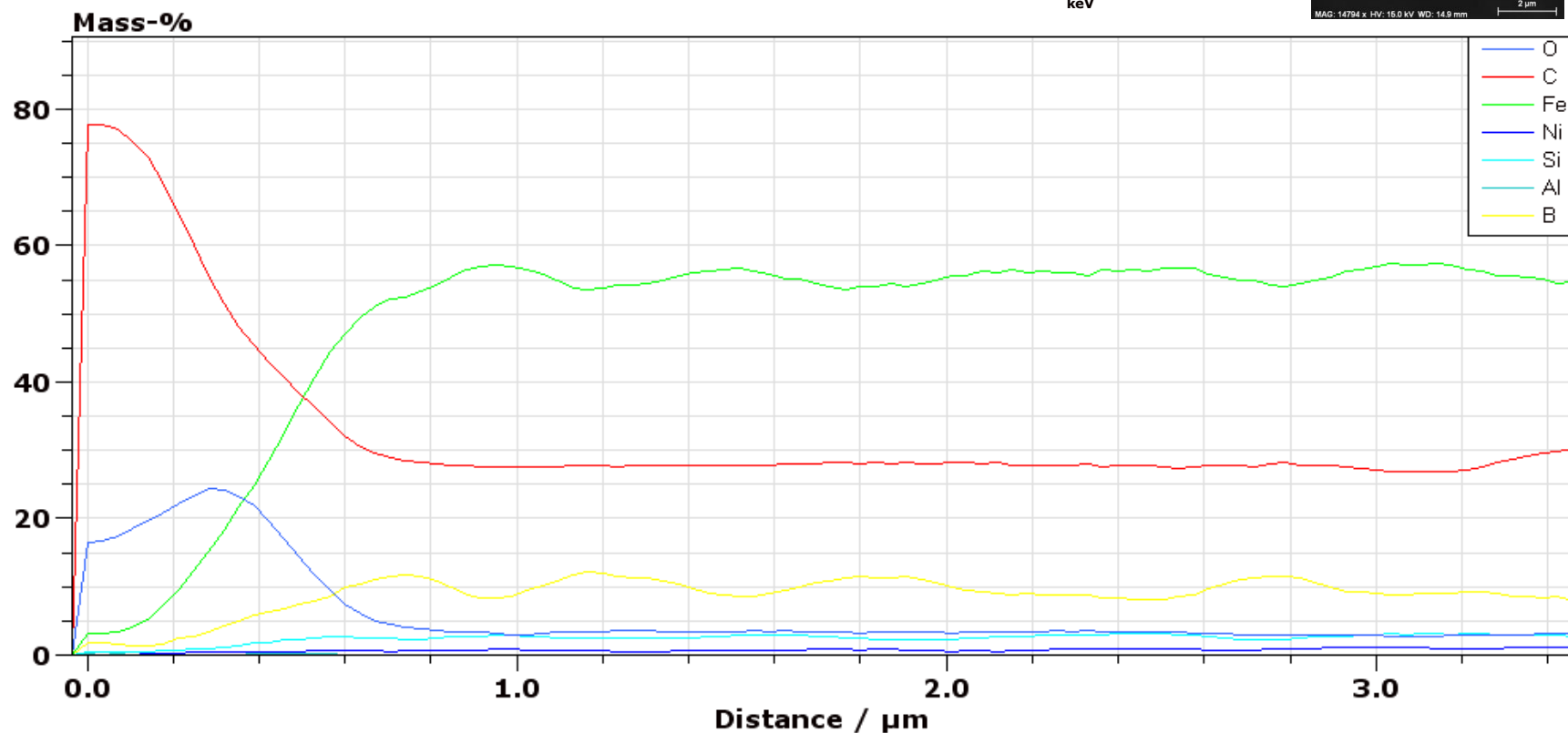
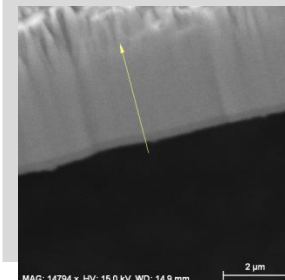
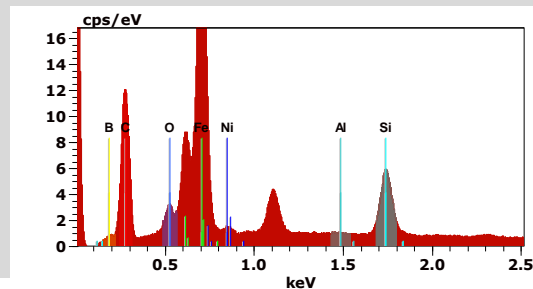
FIB analiza za odgrevan uzorak



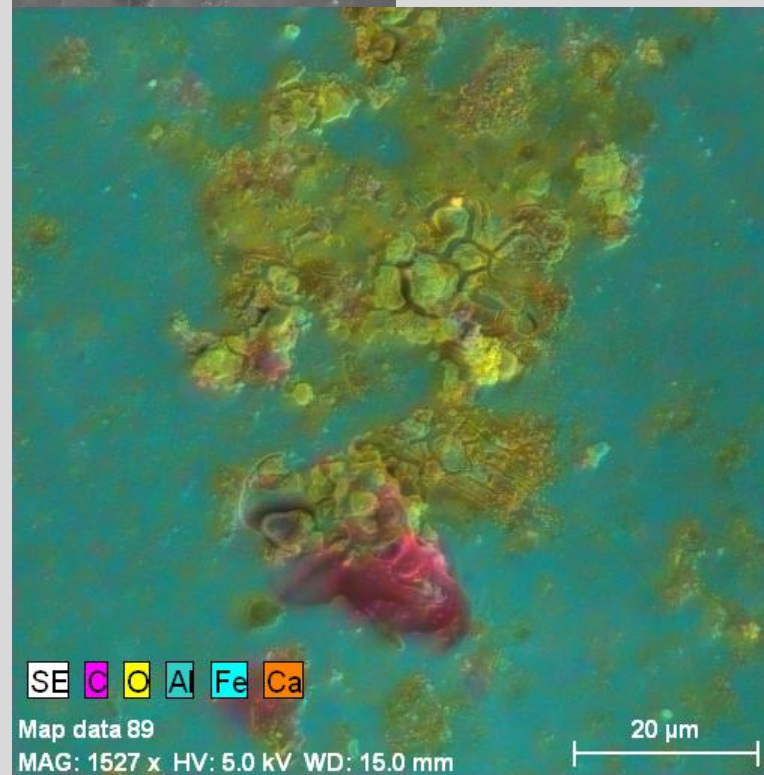
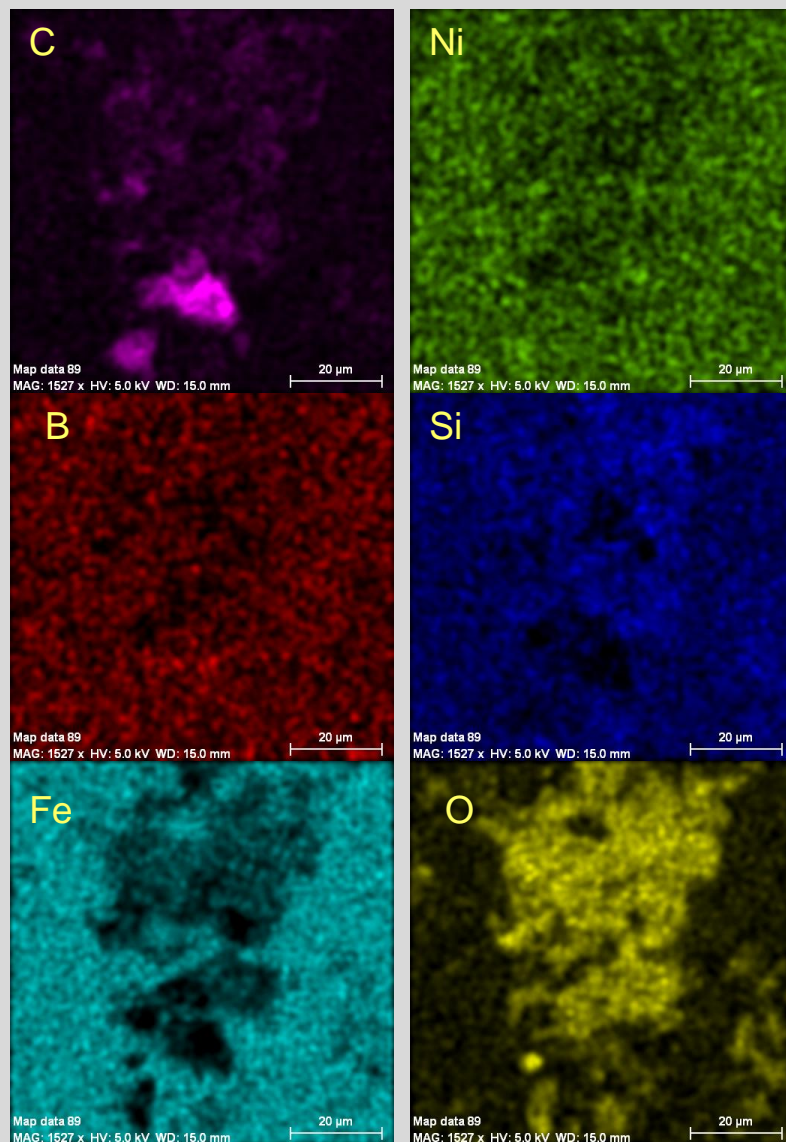
SEM HV: 15 kV	WD: 14.88 mm	 2 μm	MIRA3 TESCAN  PERFORMANCE IN NANOSPACE
View field: 8.14 μm	Det: BSE		
SEM MAG: 53.2 kx	SM: RESOLUTION		



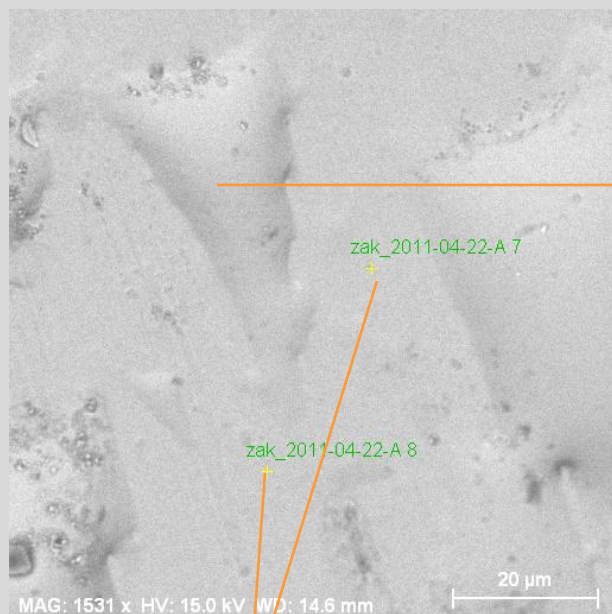
Promena hemijskog sastava od površine zagrejanog uzorka



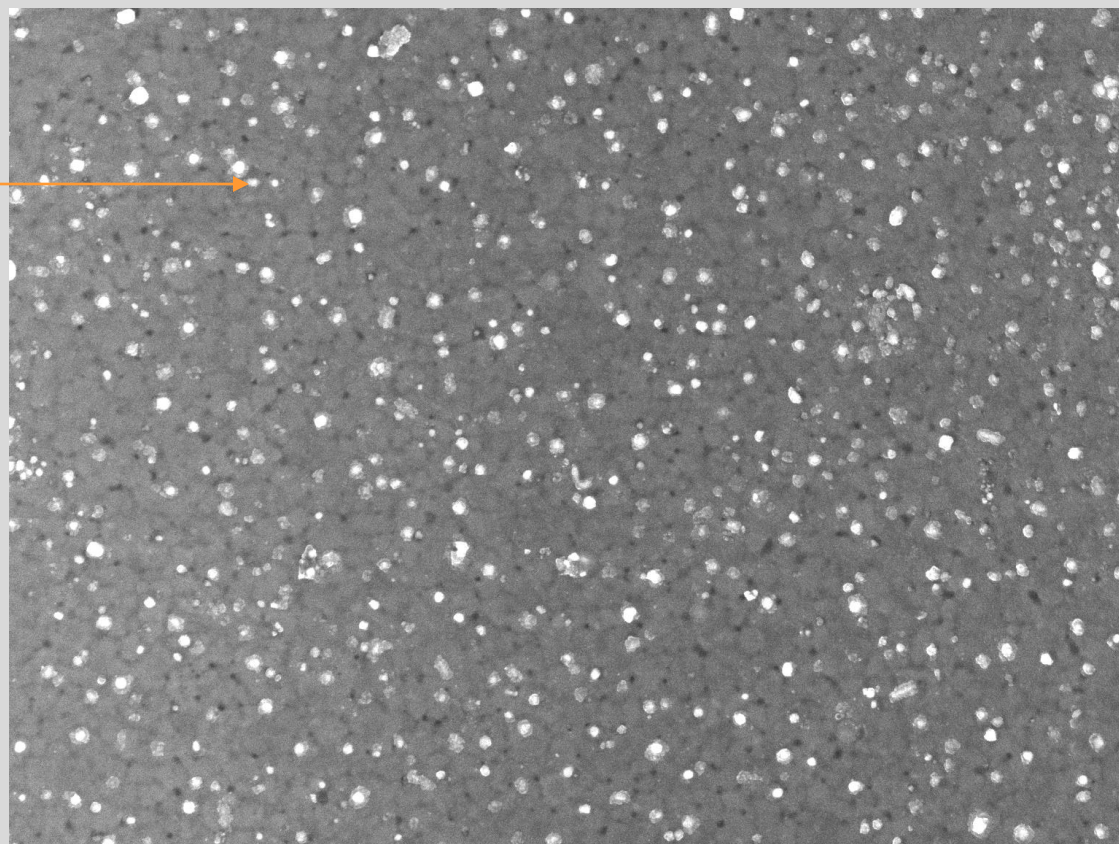
Odgrevan uзорak- chemical map of the „corrosion product“




Morfologija odgrevanog uzorka



Results of the measurement of the chemical analysis are contained on the next page



SEM HV: 15 kV	WD: 5.06 mm	 2 µm	MIRA3 TESCAN
View field: 12.7 µm	Det: SE		 TESCAN PERFORMANCE IN NANOSPACE
SEM MAG: 34.1 kx	SM: RESOLUTION		

Summary of the chemical analysis

Mass percent (%)

Spectrum		B	C	N	O	Al	Si	S	Ca	Fe	Co	Ni	Ga

zak_2011-04-22	1	1.21	5.60	-	0.70	0.45	4.79	-	-	85.25	-	2.00	-
zak_2011-04-22	2	2.80	1.86	-	0.67	0.00	4.39	-	-	88.76	-	1.51	-
zak_2011-04-22	3	2.64	2.67	-	1.18	0.03	4.37	-	-	87.22	-	1.88	-
zak_2011-04-22	4	3.73	3.37	-	1.29	0.06	4.45	-	-	85.72	-	1.39	-
zak_2011-04-22	5	7.16	4.48	-	0.89	0.06	4.26	-	-	81.76	-	1.39	-
zak_2011-04-22	6	4.89	5.16	-	2.88	0.05	4.14	-	-	81.48	-	1.38	-
zak_2011-04-22	7	5.64	2.06	-	1.05	0.02	4.48	-	-	84.88	-	1.87	-
zak_2011-04-22	8	0.00	1.51	-	0.80	0.05	4.63	-	-	91.34	-	1.66	-
zak_2011-04-22	9	3.73	6.33	-	2.20	-	5.91	-	-	80.44	-	1.38	-
zak_2011-04-22-A	1	2.13	1.13	-	4.49	0.03	9.89	-	-	80.99	-	0.80	0.55
zak_2011-04-22-A	2	4.56	1.75	-	5.28	0.19	7.81	-	-	76.03	-	2.17	2.21
zak_2011-04-22-A	3	3.44	1.06	-	4.99	0.00	8.21	-	-	79.10	-	1.36	1.83
zak_2011-04-22-A	4	1.05	9.75	0.94	25.22	0.11	4.44	0.73	9.64	38.03	5.94	0.74	3.41
zak_2011-04-22-A	5	1.96	2.51	0.61	17.21	0.11	12.01	-	6.51	56.46	0.31	0.78	1.52
zak_2011-04-22-A	6	0.99	3.13	0.85	41.81	0.84	14.55	-	6.49	27.85	0.23	0.06	3.21
zak_2011-04-22-A	7	1.53	1.91	0.00	2.35	0.17	5.14	-	0.29	85.59	1.39	1.63	0.00
zak_2011-04-22-A	8	1.69	2.00	0.00	1.81	0.25	5.24	-	0.02	86.14	2.09	0.76	0.00

Mean value:		2.89	3.31	0.48	6.76	0.15	6.40	0.73	4.59	76.30	1.99	1.34	1.59
Sigma:		1.89	2.30	0.45	11.20	0.22	3.10	0.00	4.25	18.09	2.34	0.55	1.34
Sigma mean:		0.46	0.56	0.11	2.72	0.05	0.75	0.00	1.03	4.39	0.57	0.13	0.32

Chemical analysis of the as-prepare sample

Chemical analysis of the annealed sample

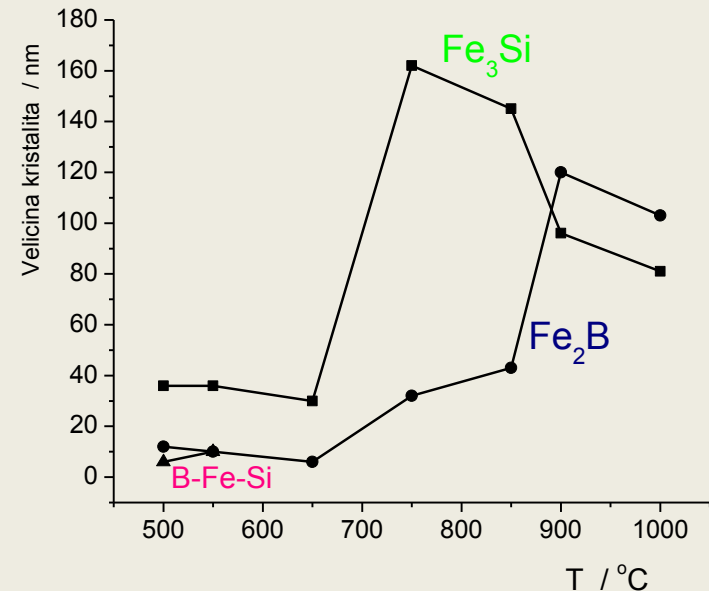
The presence of the Ga in the annealed sample is a consequence of the using of the FIB

Mikrostrukturni parametri

Temperatura °C	Fe ₃ Si nm	Fe ₂ B nm	Fe ₃ B nm
500	36	12	6
550	36	10	10
650	30	6	-
750	162	32	
850	145	43	
900	96	120	
1000	81	103	

Veličina kristalita nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

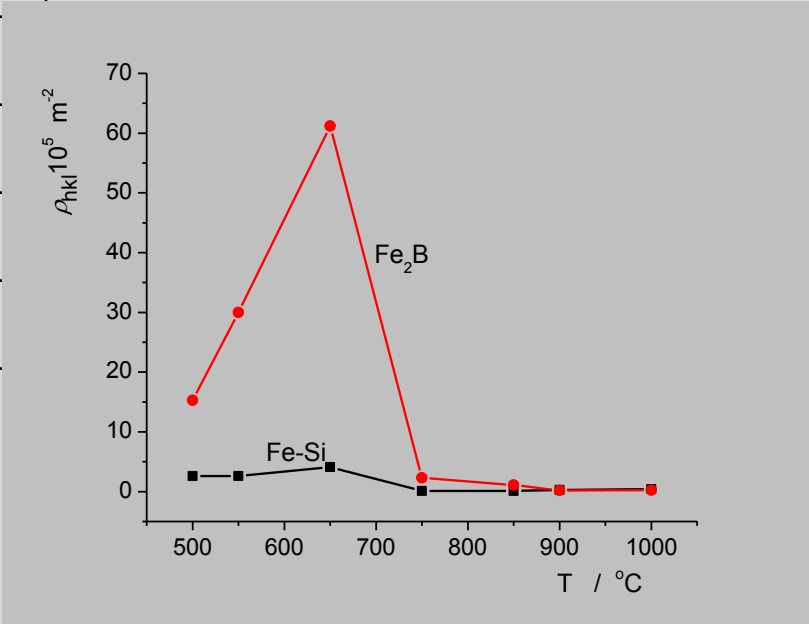
$$D_{hkl} = \frac{k\lambda}{\psi \cos \theta}$$

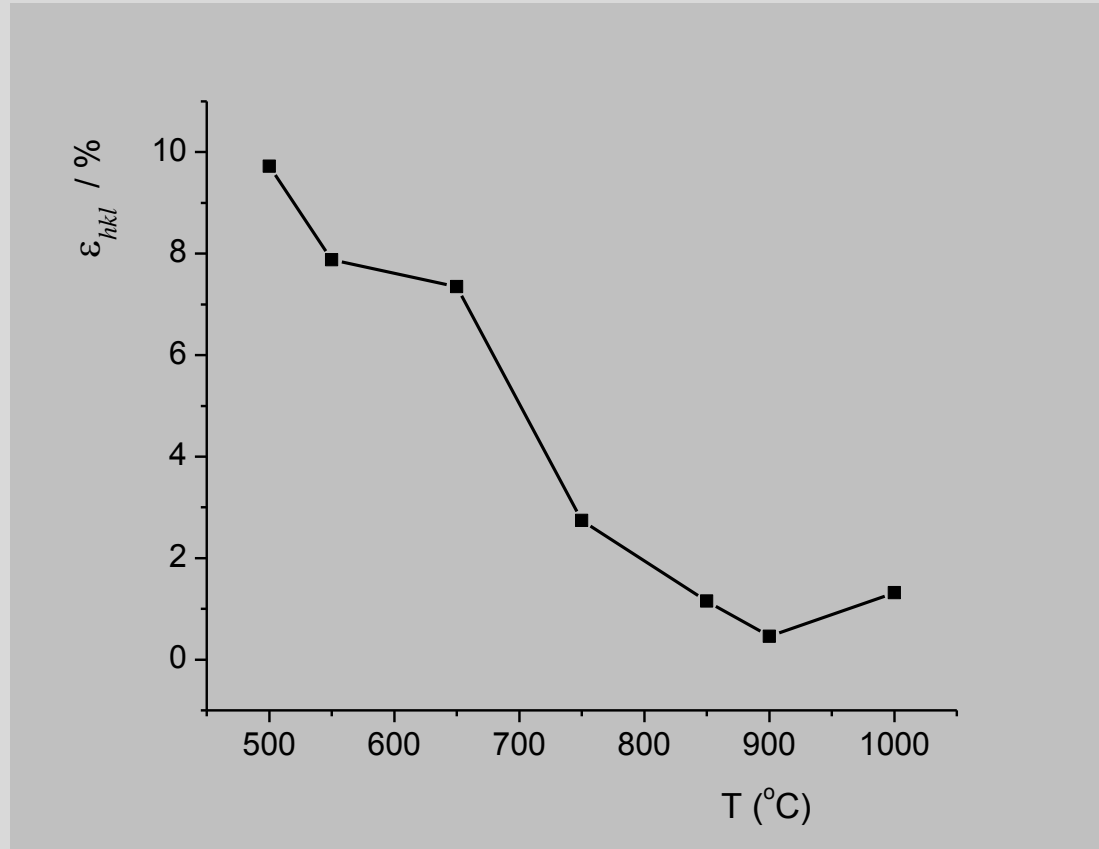


Gustina dislokacija nađenih faza u uzorcima legure odgrevanim na raznim temperaturama.

Temper. °C	ρ_{hkl}^{-2} m	ρ_{hkl}^{-2} m	ρ_{hkl}^{-2} m	ϵ_{hkl} %
	Fe ₃ Si	Fe ₂ B	Fe ₃ B	
500	2,6 10 ¹⁵	1,53 10 ¹⁶	3,70 10 ¹⁶	9,72
550	2,6 10 ¹⁵	3,00 10 ¹⁶	1.78 10 ¹⁶	7,88
650	4,12 10 ¹⁵	6,12 10 ¹⁶	-	7,35
750	0,11 10 ¹⁵	0,23 10 ¹⁶	-	2,74
850	0,28 10 ¹⁵	0,11 10 ¹⁶	-	1,15
900	0,29 10 ¹⁵	0,02 10 ¹⁶	-	0,46
1000	0,40 10 ¹⁵	0,03 10 ¹⁶	-	1,32

$$\rho_{hkl} = \frac{3}{D_{hkl}^2}$$





Promena mikronaprezanja sa temperaturom odgrevanja uzoraka legure.

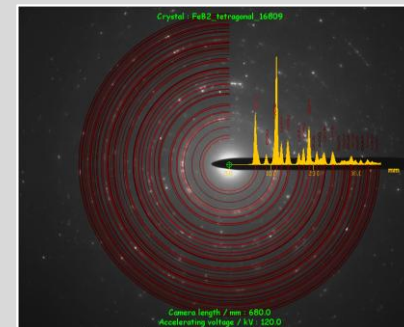
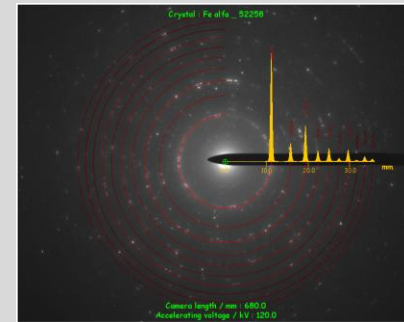
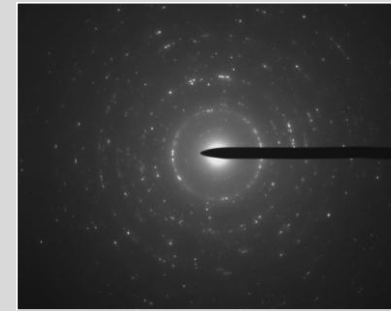
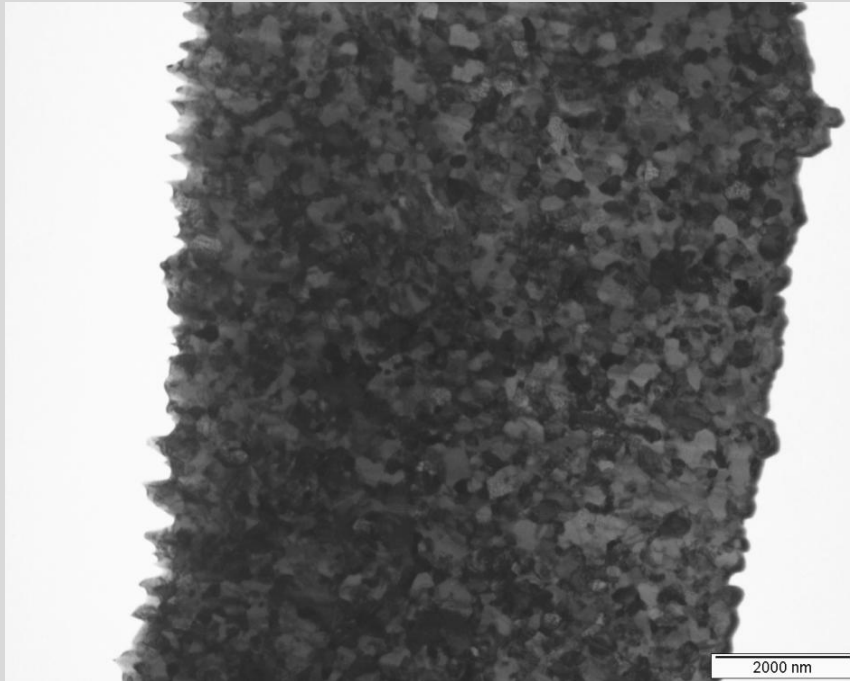


Fig. 5. Overview of the lamella (the surface of the sheet is on its right-hand side) together with the SAED pattern; $\alpha\text{Fe}(\text{Si})$ (middle) and tetragonal phase of Fe_2B (bottom) were identified.

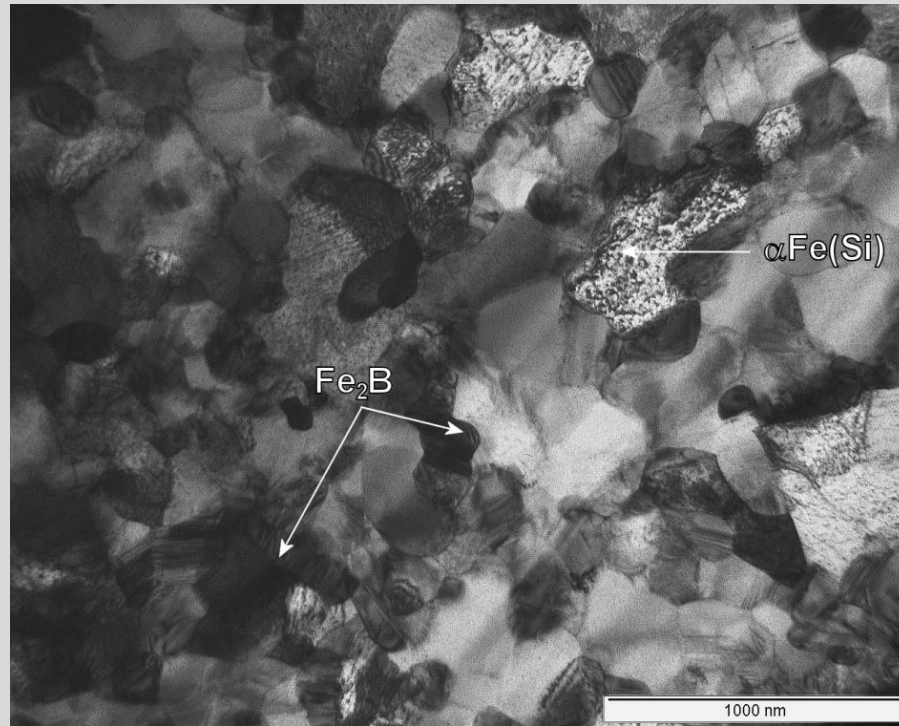


Fig. 6. Lamella structure with marked grains of phases identified using dark field imaging.

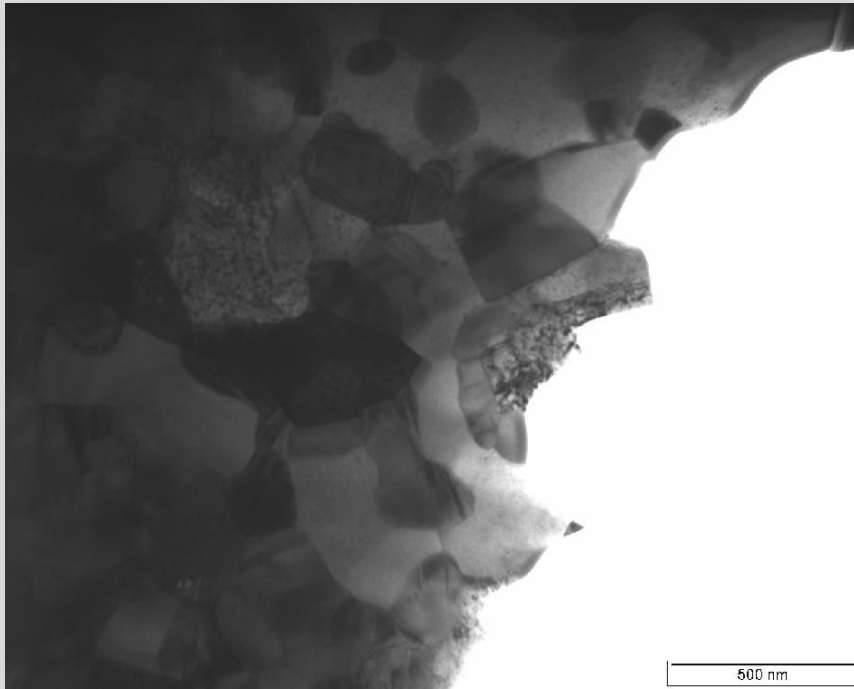


Fig 7 Bright field and dark field images of the same sample area from the side opposite to the surface show the grain of the $\alpha\text{Fe(Si)}$ phase (left) – DF in (1 -1 0) spot (right)

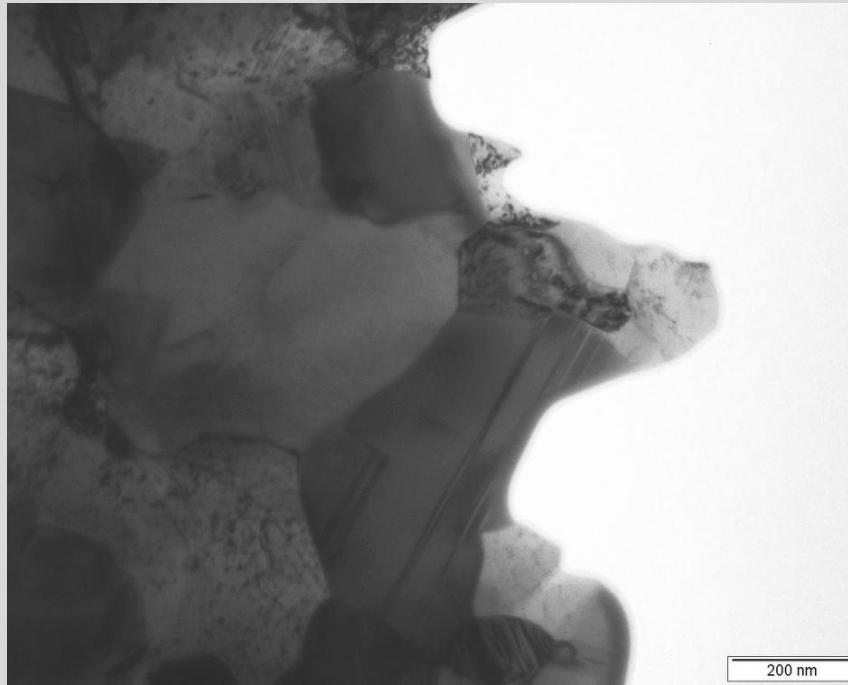
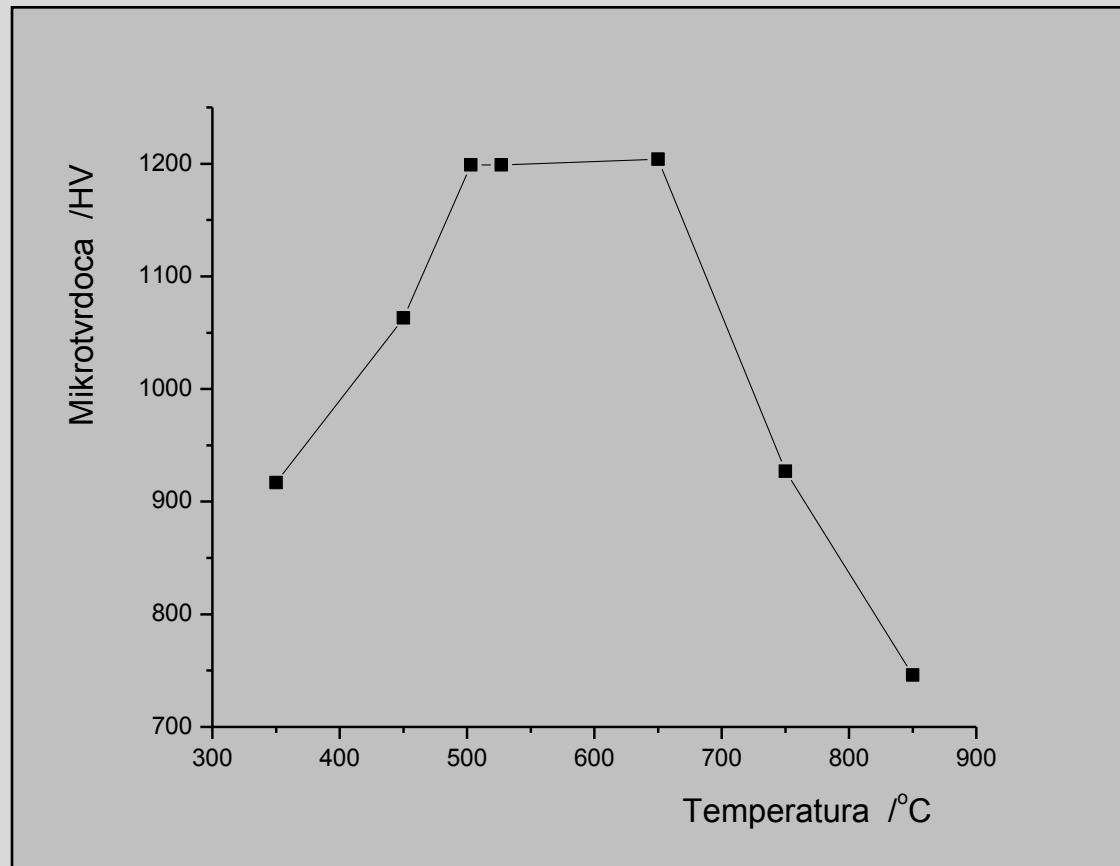


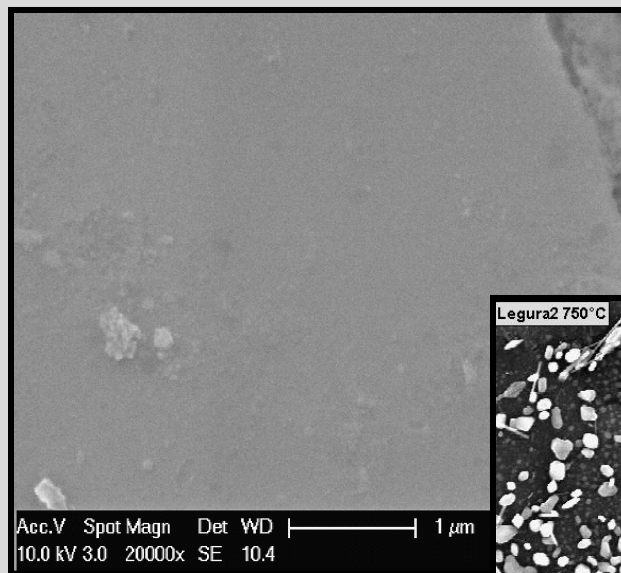
Fig. 8. Detail of grain structure of the sample and their morphology at the side opposite to the surface.

Uticaj strukturnih promena na mikrotvrdoću

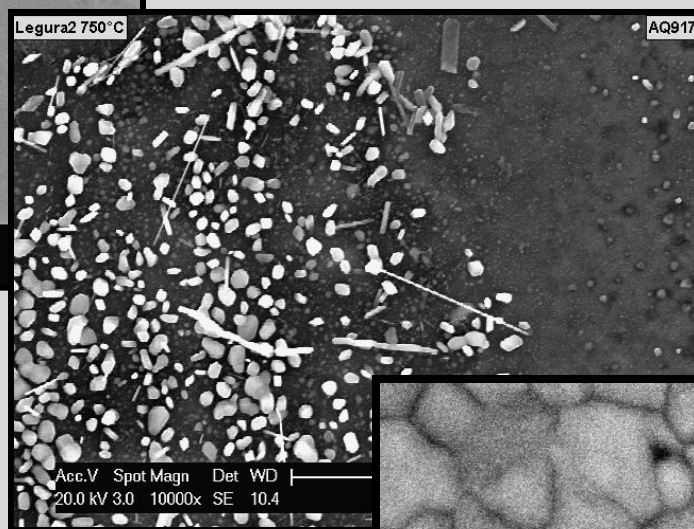


Temperaturska zavisnost mikrotvrdoće uzoraka legure odgrevanih na raznim temperaturama.

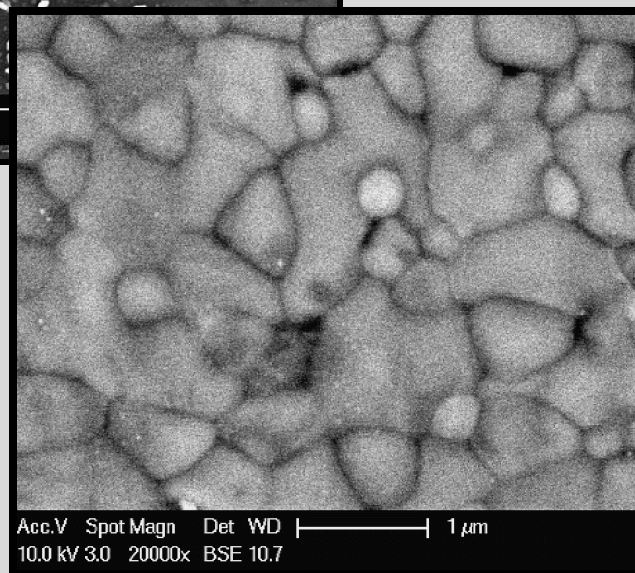
Morfologija površine



Polazna



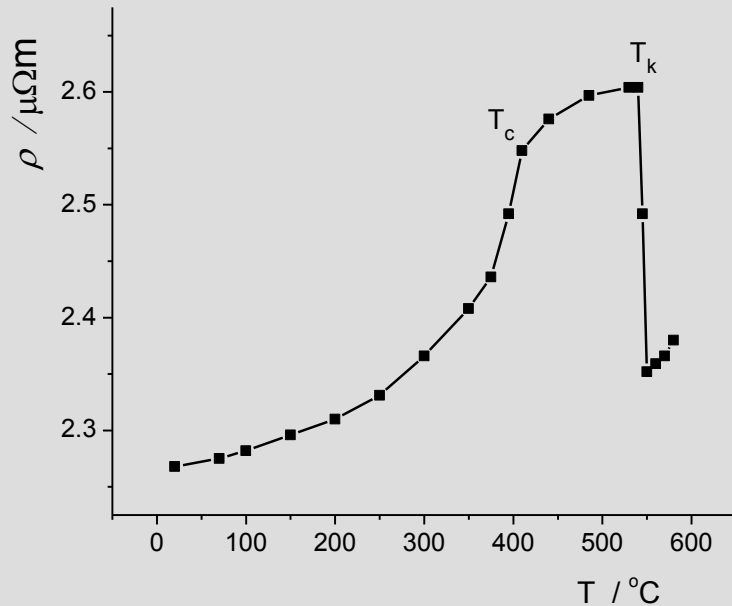
850



1000

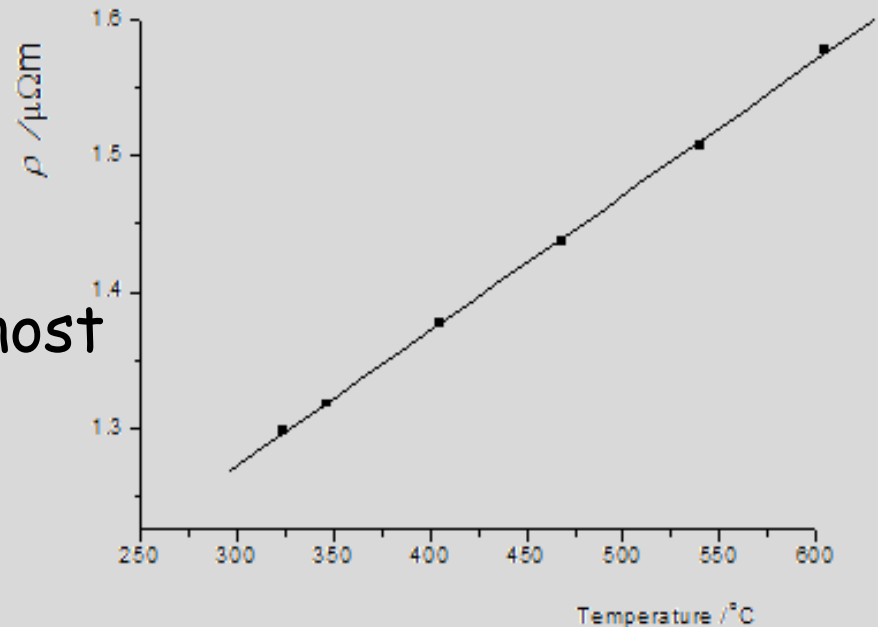
SEM mikgrografije površine
polazne legure i uzoraka legure
odgrejanih na 850 i 1000°C.

Uticaj strukturnih promena na električna svojstva

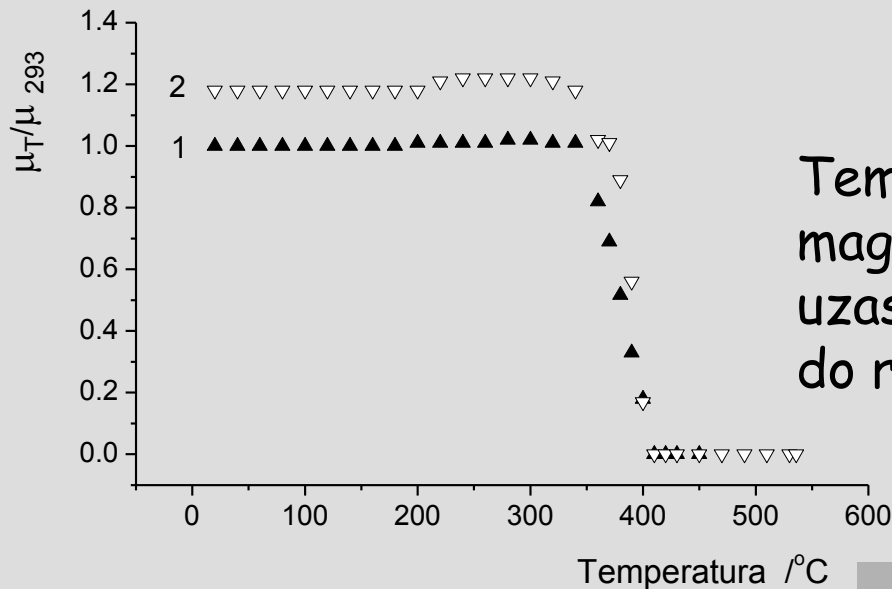


Temperaturska zavisnost
električne otpornosti
polazne legure.

Temperaturska zavisnost
električne otpornosti
iskristalisale legure.

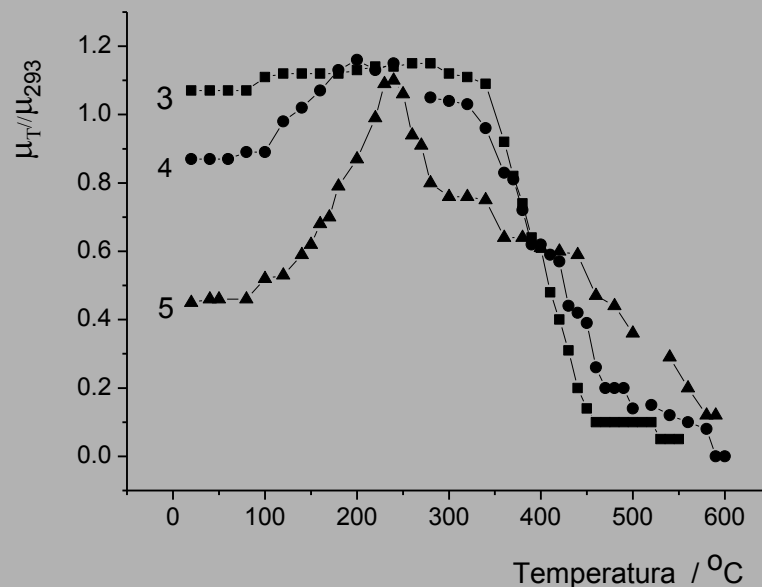


Uticaj strukturnih promena na magnetna svojstva

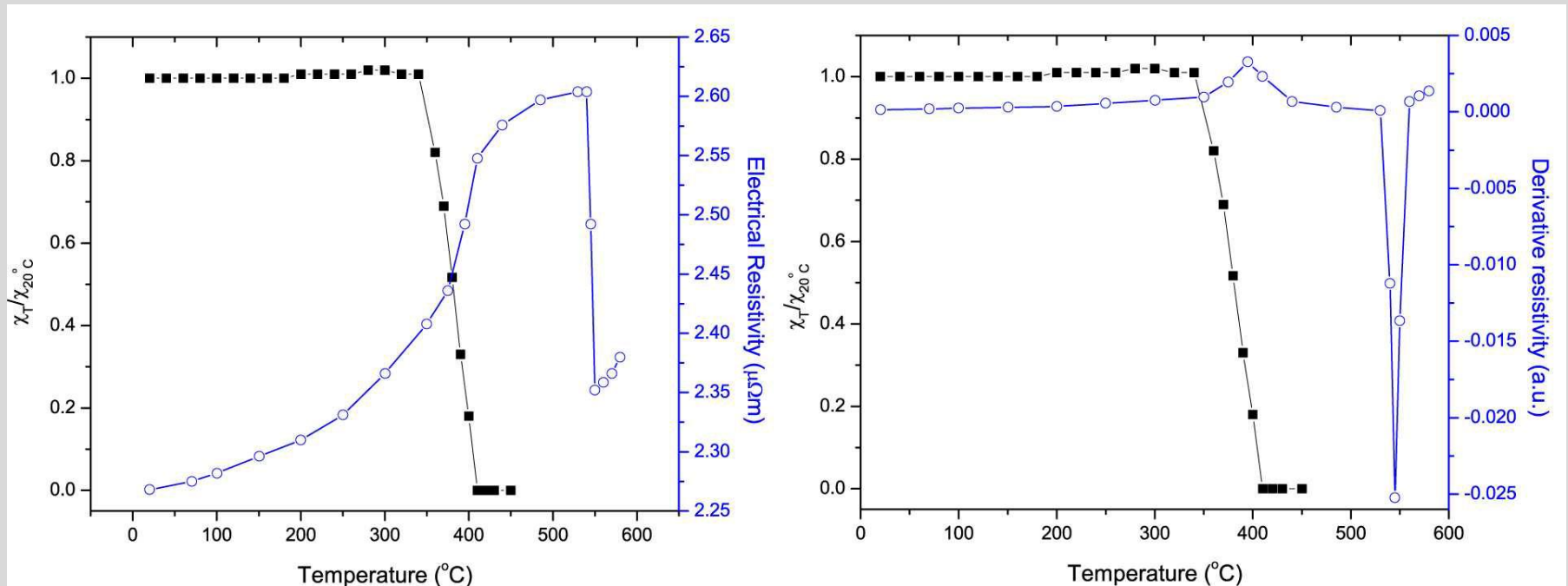


Temperaturska zavisnost relativne magnetne susceptibilnosti tokom dva uzastopna ciklusa zagrevanja legure do različitih temperatura.

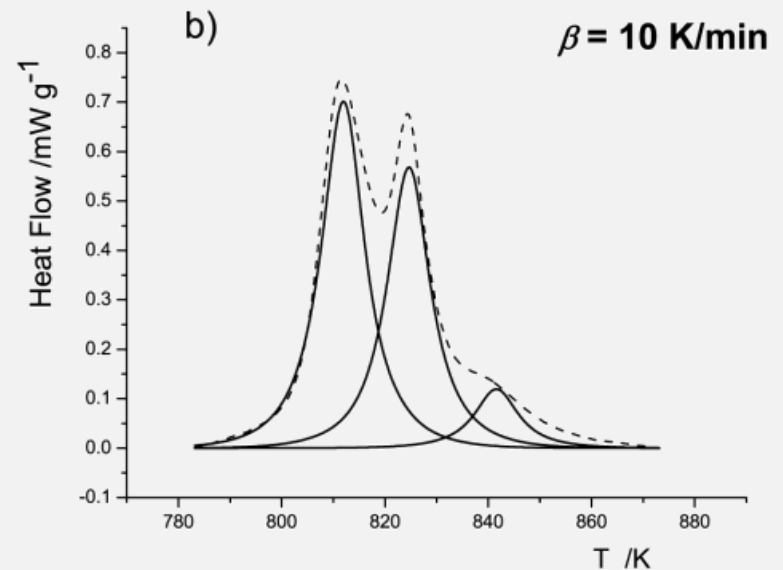
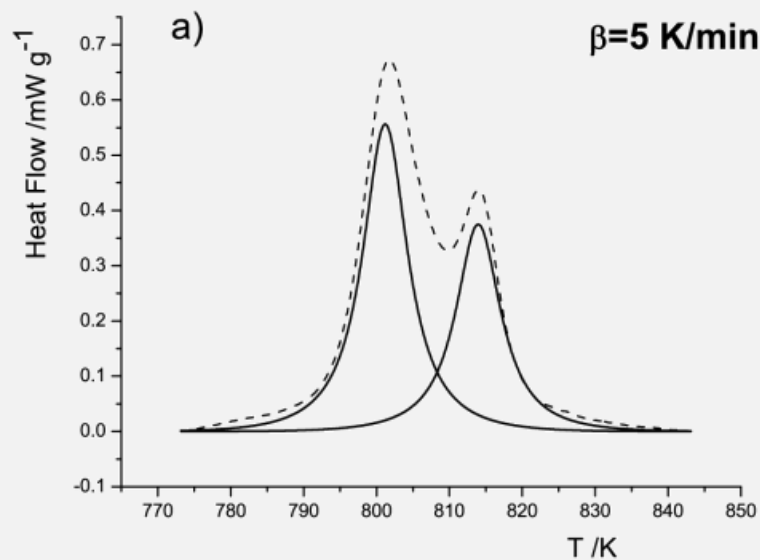
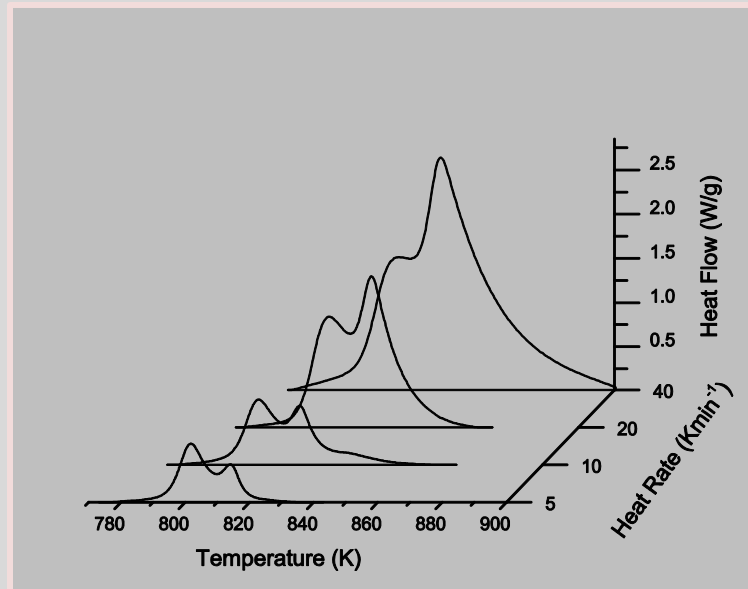
Temperaturska zavisnost relativne magnetne susceptibilnosti za treći, četvrti i peti ciklus zagrevanja legure do različitih temperatura.

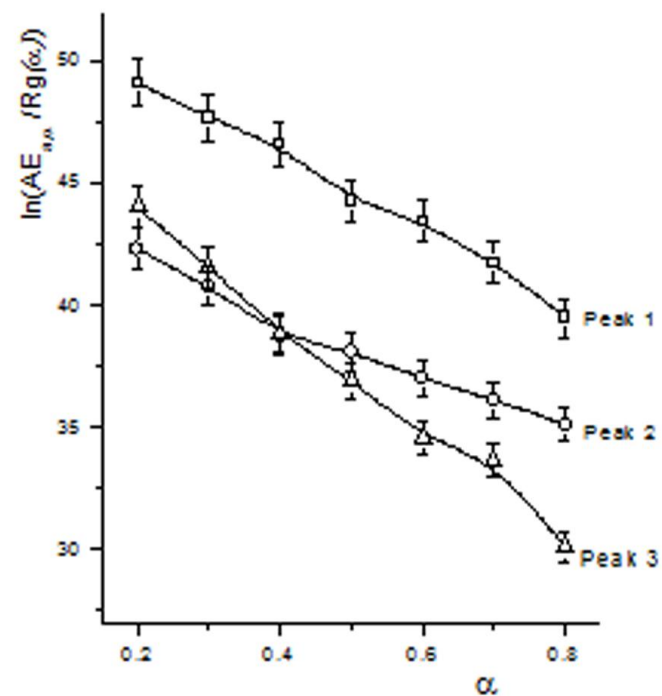
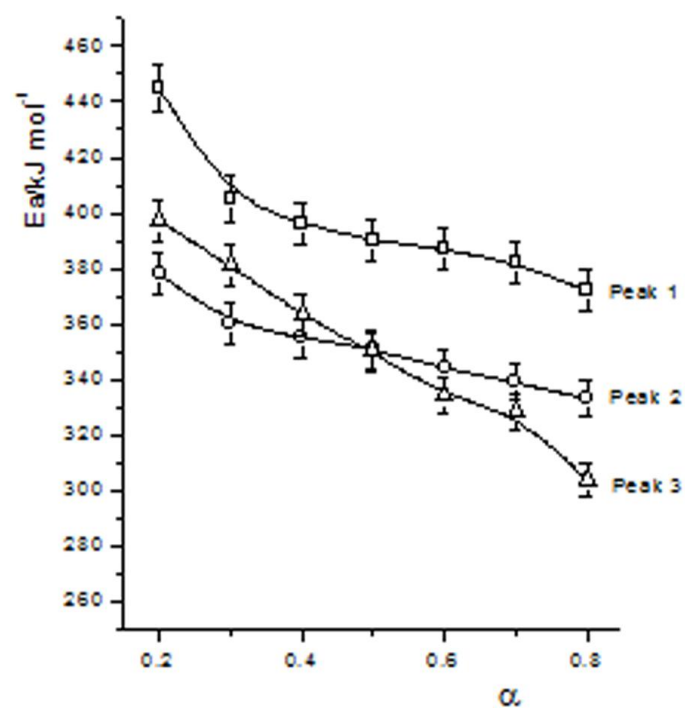


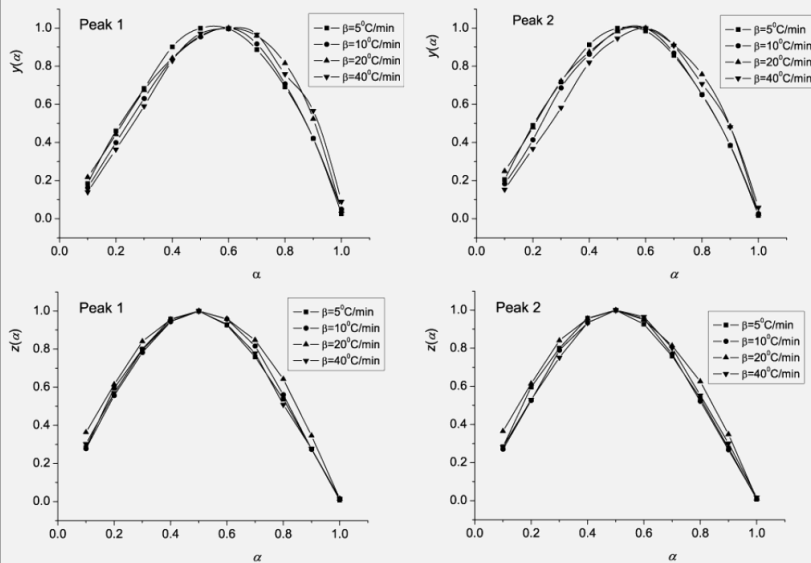
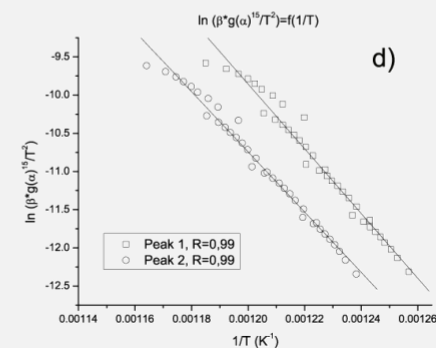
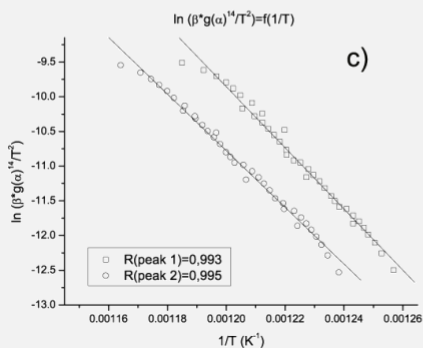
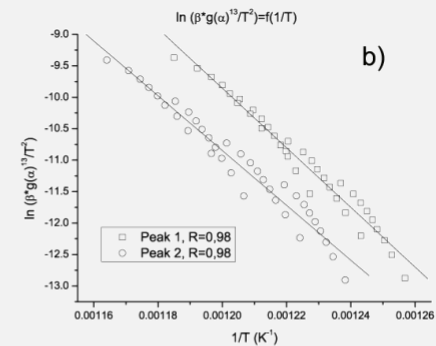
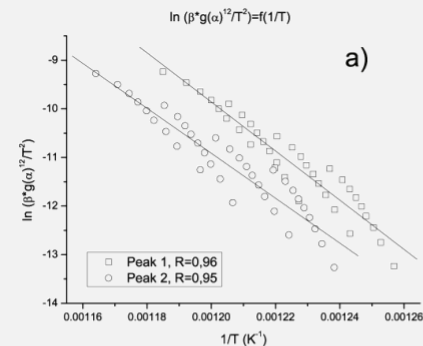
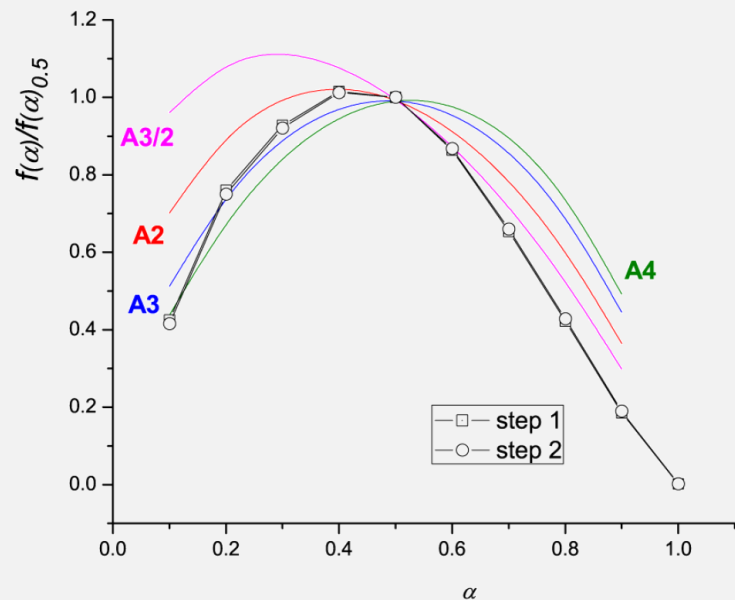
Temperaturska zavisnost magnetne provodljivosti prikazana sa temperatuirskom zavisnošću električne provodljivosti



Mehanizam i kinetika kristalizacije







Step 1:

$$g(\alpha) = [-\ln(1-\alpha)]^{1/3}; f(\alpha) = 3(1-\alpha)[- \ln(1-\alpha)]^{2/3}$$

$$E_a = 375.1 \pm 0.8 \text{ kJ/mol i } \ln A = 56.2 \pm 1.0$$

Step 2:

$$g(\alpha) = [-\ln(1-\alpha)]^{1/3}; f(\alpha) = 3(1-\alpha)[- \ln(1-\alpha)]^{2/3}$$

$$E_a = 341.6 \pm 0.5 \text{ kJ/mol i } \ln A = 49.3 \pm 0.5$$

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D. M. Minić, A. Gavrilović, P. Angerer, D.G. Minić, A. Marčić, *Structural Transformations of $Fe_{75}Ni_2Si_{13}C_2$ Amorphous Alloy Induced by Thermal Treatment*, Journal of Alloys and Compounds 476(2009)705-709.

D M. Minić, A. Maričić, *Influence of heating on electric and magnetic properties of $Fe_{75}Ni_2B_{13}Si_8C_2$ amorphous alloy*, Materials Science Engineering B 172 (2010) 127-131.

D.G. Minić, V.A. Blagojević, Lj.E. Mihajlović, V.R. Ćosović, D.M. Minić, *Kinetics and mechanism of structural transformations of $Fe_{75}Ni_2Si_8B_{13}C_2$ amorphous alloy induced by thermal treatment*, Thermochimica Acta, 519(2011) 83-89

V.A. Blagojević, D.M. Minić, T. Žák, D.M. Minić, *Influence of thermal treatment on structure and microhardness of $Fe_{75}Ni_2Si_8B_{13}C_2$ amorphous alloy*, Intermetallics 19(2011)1780-1785.

Dušan M. Minić, Vladimir A. Blagojević, Aleksa M. Maričić, Tomáš Žák, Dragica M. Minić, *Influence of structural transformations on functional properties of $Fe_{75}Ni_2Si_8B_{13}C_2$ amorphous alloy*, Mater. Phys.Chem.134(1)(2012)111–115.

Dušan M. Minić, Vladimir A. Blagojević, Bohumil David, Nadezda Pizurova, Tomáš Žák, Dragica M. Minić, *Influence of thermal treatment on microstructure of $Fe_{75}Ni_2Si_8B_{13}C_2$ amorphous alloy*, Intermetallics 25 (2012) 75-79.

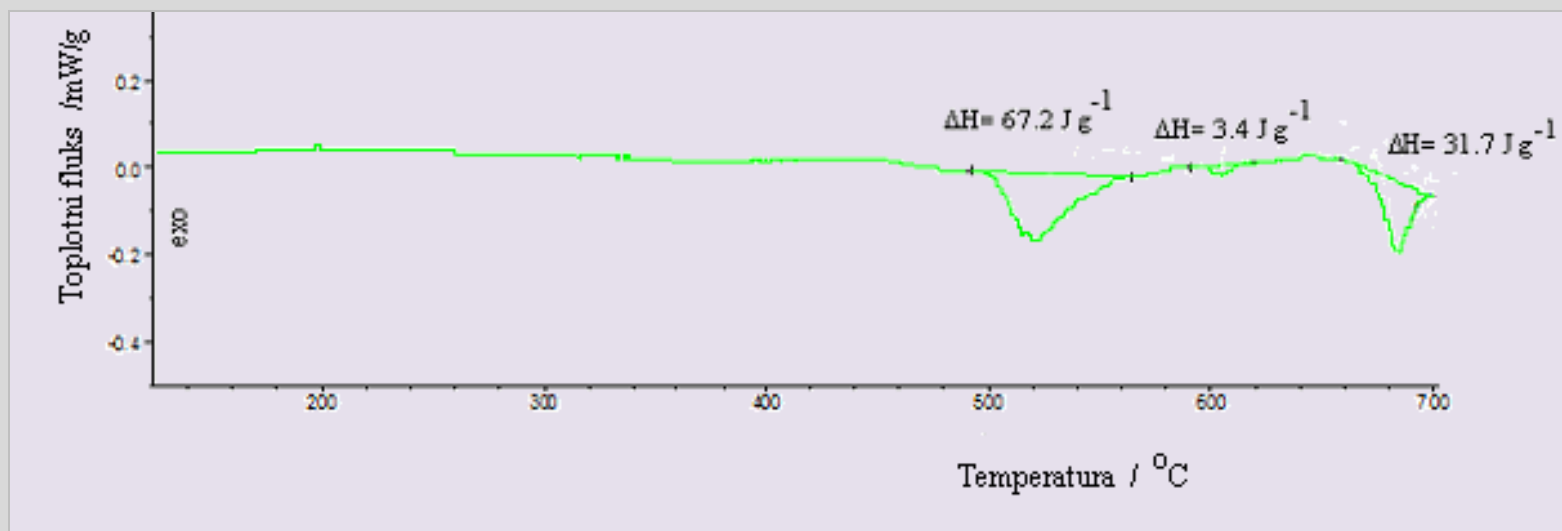
Dragica M. Minić, Vladimir A. Blagojević, Dušan M. Minić, Bohumil David, Naděžda Pizúrová, Tomáš Žák, *Crystal growth of iron nanorods in thermally treated $Fe_{75}Ni_2Si_8B_{13}C_2$ amorphous alloy*, Metallurgical Materials Transactions A, 43(9)(2012)3062-3609.

Dušan M. Minić, Vladimir A. Blagojević, Dragica M. Minić, *Uticaj termičkog tretmana na strukturu i svojstva amorfne legure $Fe_{75}Ni_2Si_8B_{13}C$* , Hem. Ind. 66 (5) (2012) 769–779

Vladimir A. Blagojević, Milica Vasić, Dušan M. Minić, Dragica M. Minić, *Kinetics and thermodynamics of thermally induced structural transformations of amorphous $Fe_{75}Ni_2Si_8B_{13}C_2$ alloy*, Thermochemica Acta, 549 (2012) 35-41.

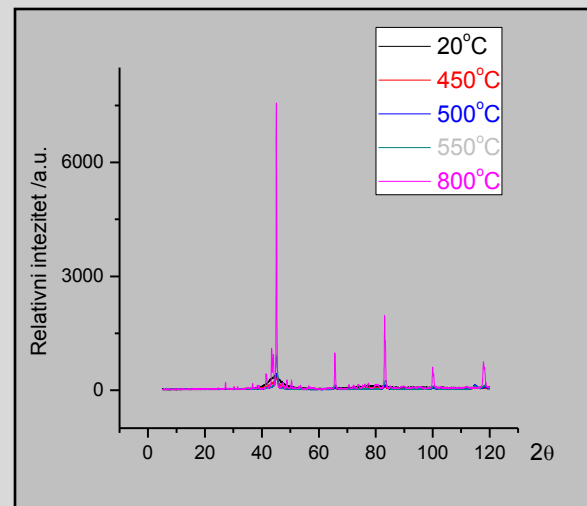
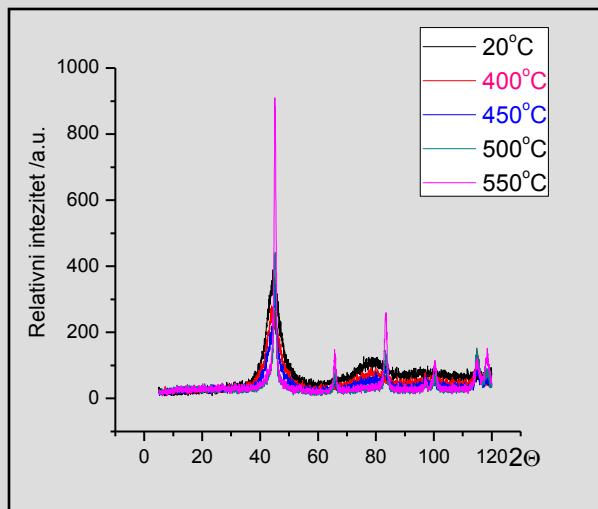
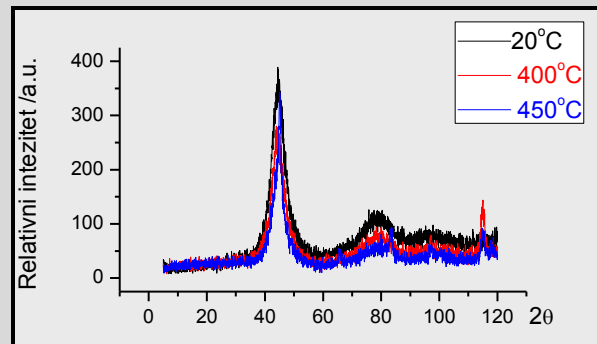
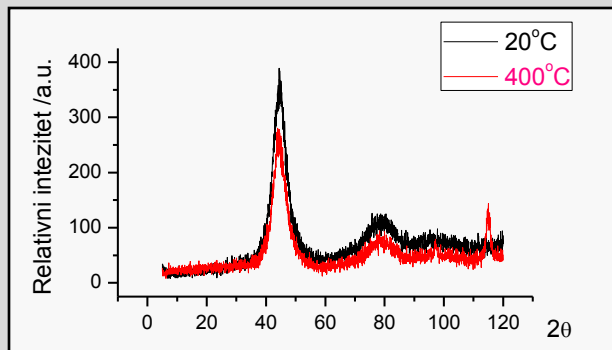
Amorfna $\text{Fe}_{73,5}\text{Cu}_1\text{Nb}_3\text{Si}_{15,5}\text{B}_7$ legura

Termička stabilnost



DSC polazne legure pri brzini zagrevanja 4 °C/min.

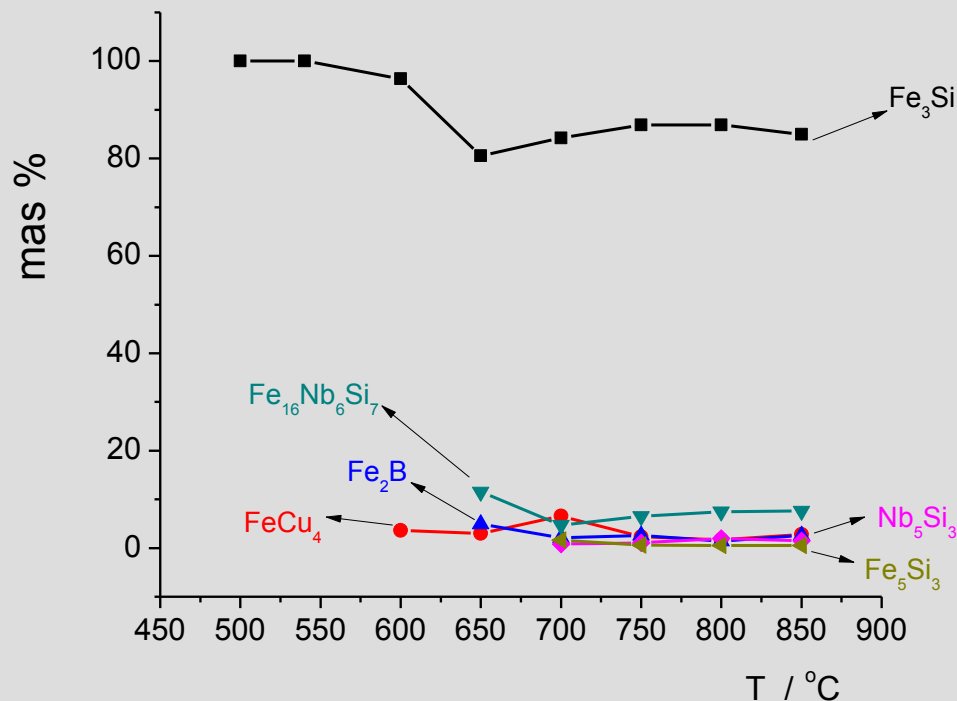
Strukturne transformacije



Difraktogrami X-zraka za polaznu leguru i uzorke legure koji su odgrevani na raznim temperaturama

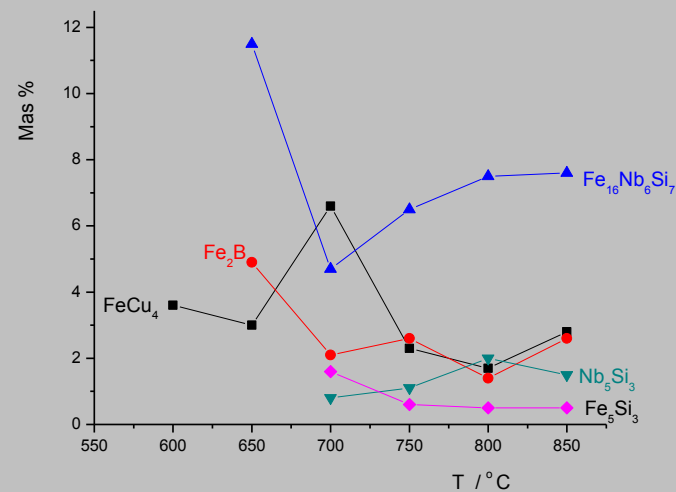
Kristalna simetrija, prostorna grupa (Hermann-Mauguin) i parametri rešetke nađenih faza u iskristalisanjoj leguri

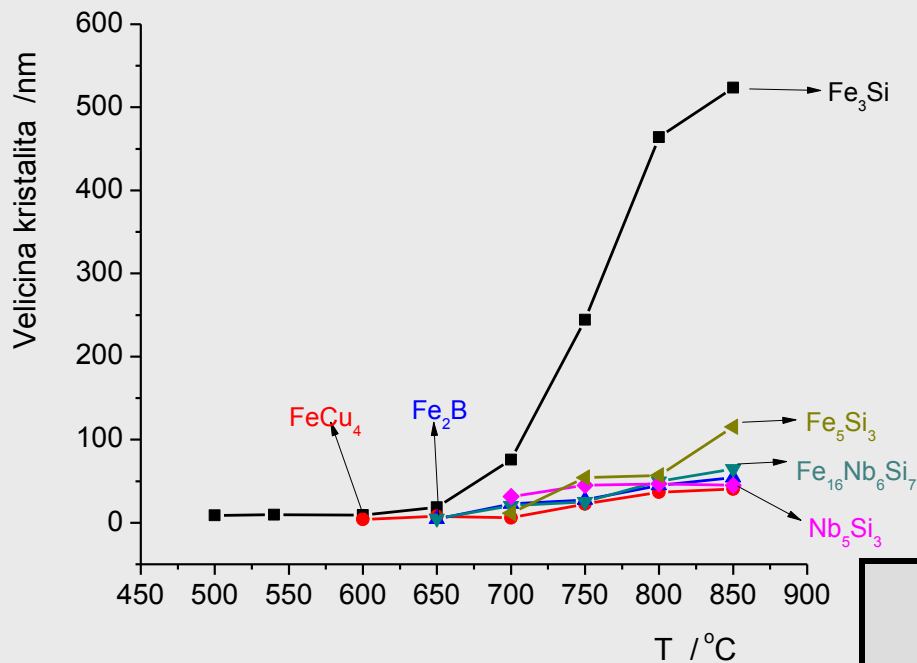
Faza	Prostorna grupa	Kristalni sistem	Parametri rešetke [Å]	
			<i>a</i>	<i>c</i>
Fe ₃ Si [ICDD-PDF 03-065-0146]	Fm-3m	Kubni	5.670	-
FeCu ₄ [ICDD-PDF 03-065-7002]	Fm-3m	Kubni	3.618	-
Fe ₁₆ Nb ₆ Si ₇ [ICDD-PDF 00-053-0459]	Fm-3m	Kubni	11.338	-
Fe ₂ B [ICDD-PDF 00-036-1332]	I4/mcm	Tetragonalni	5.110	4.249
Nb ₅ Si ₃ [ICDD-PDF 03-065-2785]	I4/mcm	Tetragonalni	10.018	5.072
Fe ₅ Si ₃ [ICDD-PDF 03-065-3593]	P63/mcm	Heksagonalni	6.755	4.717



Fazni sastav uzoraka
legure u funkciji
temperature odgrevanja

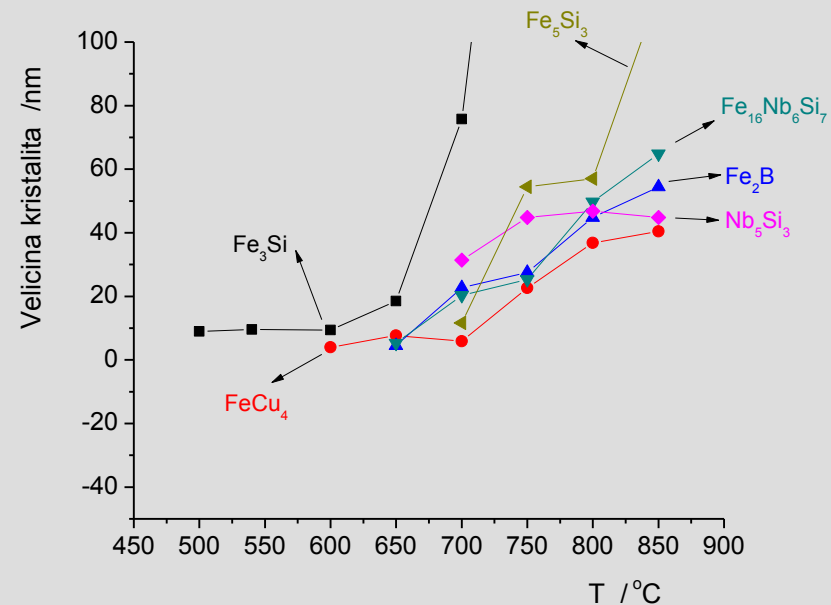
Fazni sastav manje zastupljenih
faza uzoraka legure u funkciji
temperature odgrevanja





Veličina kristalita svih
nađenih faza u funkciji
temperature odgrevanja.

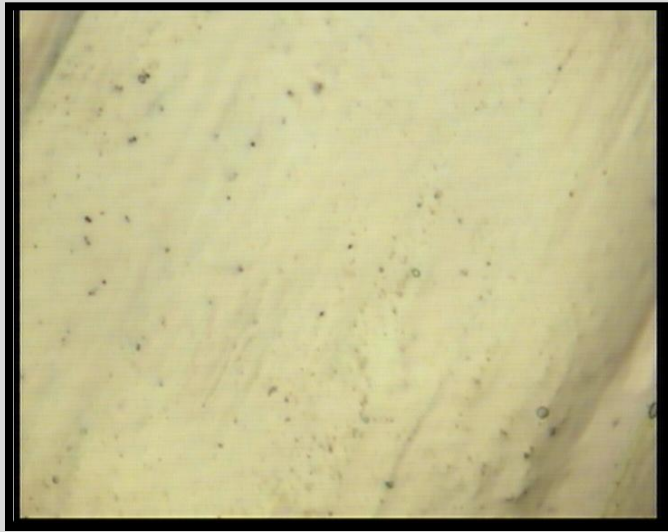
Veličina kristalita
dimenzija do 100 nm.



Gustina dislokacija faza za uzorke legure koji su odgrevani na raznim temperaturama.

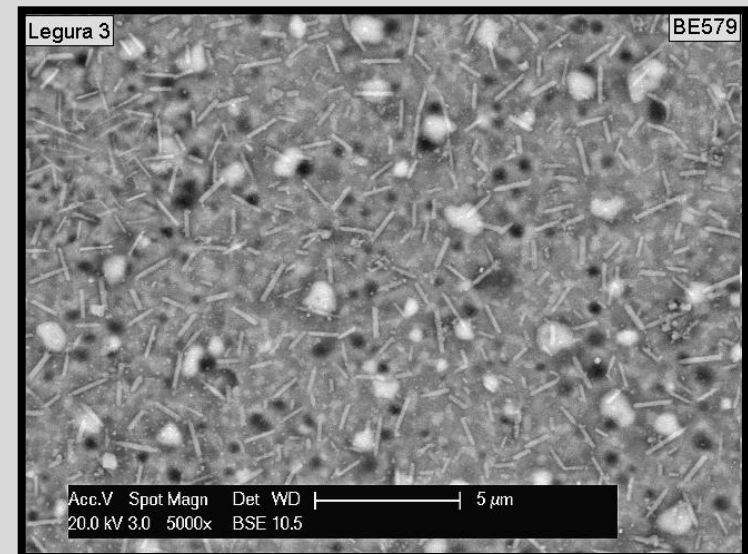
Temper. [°C]	Fe ₃ Si	FeCu ₄	Fe ₁₆ Nb ₆ Si ₇	Fe ₂ B	Nb ₅ Si ₃	Fe ₅ Si ₃
	Gustina dislokacija [m ⁻²]	Gustina dislokacija [m ⁻²]	Gustina dislokacija [m ⁻²]	Gustina dislokacija [m ⁻²]	Gustina dislokacija [m ⁻²]	Gustina dislokacija [m ⁻²]
500	$3.70 \cdot 10^{16}$	-	-	-	-	-
550	$3.26 \cdot 10^{16}$	-	-	-	-	-
600	$3.40 \cdot 10^{16}$	$1.88 \cdot 10^{17}$	-	-	-	-
650	$8.77 \cdot 10^{15}$	$5.06 \cdot 10^{16}$	$1.07 \cdot 10^{17}$	$1.48 \cdot 10^{17}$	-	-
700	$5.22 \cdot 10^{14}$	$8.62 \cdot 10^{16}$	$7.28 \cdot 10^{15}$	$5.82 \cdot 10^{15}$	$3.04 \cdot 10^{15}$	$2.23 \cdot 10^{16}$
750	$5.03 \cdot 10^{13}$	$5.87 \cdot 10^{15}$	$4.69 \cdot 10^{15}$	$3.97 \cdot 10^{15}$	$1.49 \cdot 10^{15}$	$1.01 \cdot 10^{15}$
800	$1.39 \cdot 10^{13}$	$2.22 \cdot 10^{15}$	$1.21 \cdot 10^{15}$	$1.49 \cdot 10^{15}$	$1.37 \cdot 10^{15}$	$9.23 \cdot 10^{14}$
850	$1.09 \cdot 10^{13}$	$1.84 \cdot 10^{15}$	$7.14 \cdot 10^{14}$	$1.01 \cdot 10^{15}$	$1.49 \cdot 10^{15}$	$2.25 \cdot 10^{14}$

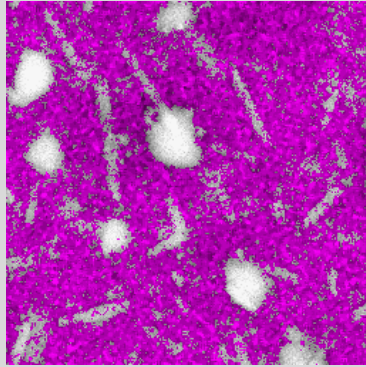
Morfologija površine



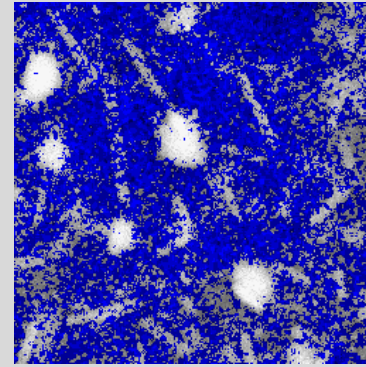
Homogena površina polazne legure

Mikrografija površine uzorka legure koji je odgrevan tokom 24h na 850°C.

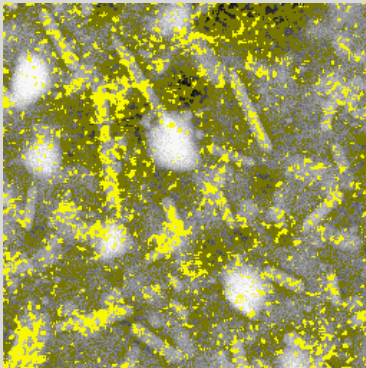




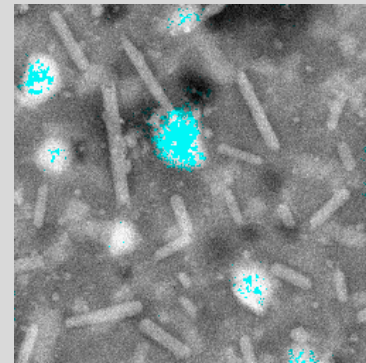
Fe



Si

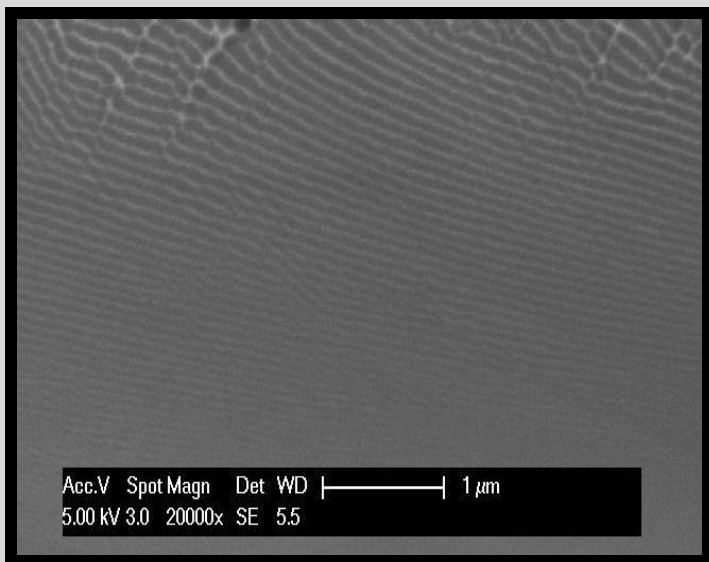


Nb



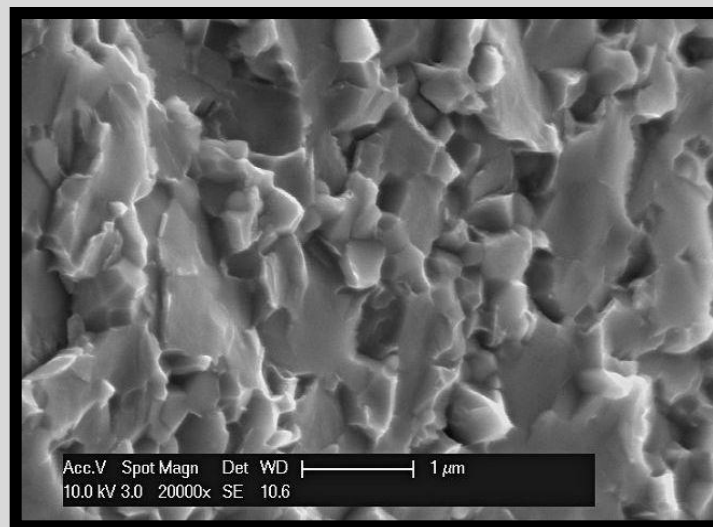
Cu

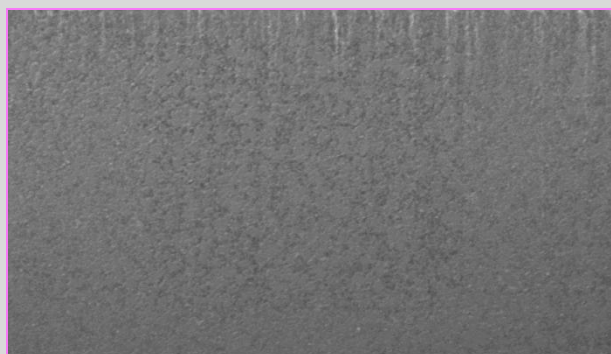
Mapiranje SEM mikrografije uzorka legure odgrejanog na 850°C radi određivanja hemijskog sastava u raznim tačkama površine.



SEM mikrofotografija
poprečnog preseka
polazne legure.

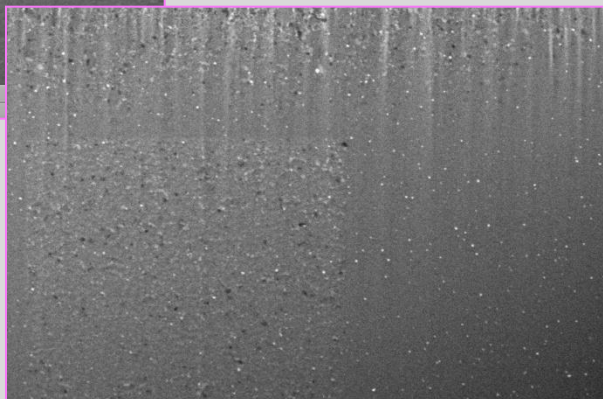
SEM mikrofotografija poprečnog
preseka uzorka legure posle
odgrevanja na 850 °C tokom 24 h.





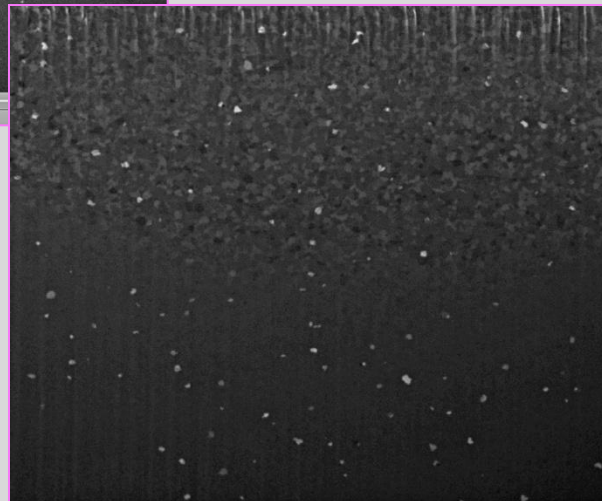
693 K

10/29/2009	HV	HFW	WD	mag	curr
10:51:00 AM	30.00 kV	13.5 μ m	30.1 mm	20000 x	30 pA



753 K

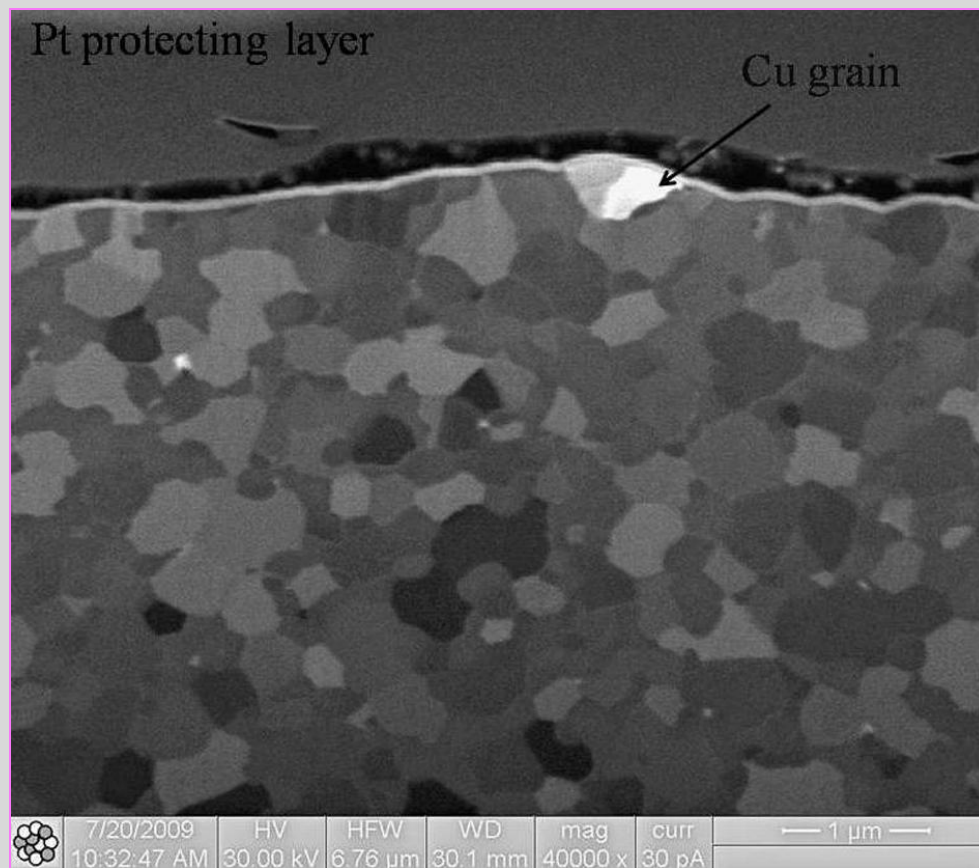
10/29/2009	HV	HFW	WD	mag	curr
3:28:50 PM	30.00 kV	13.5 μ m	30.1 mm	20000 x	30 pA



1123 K

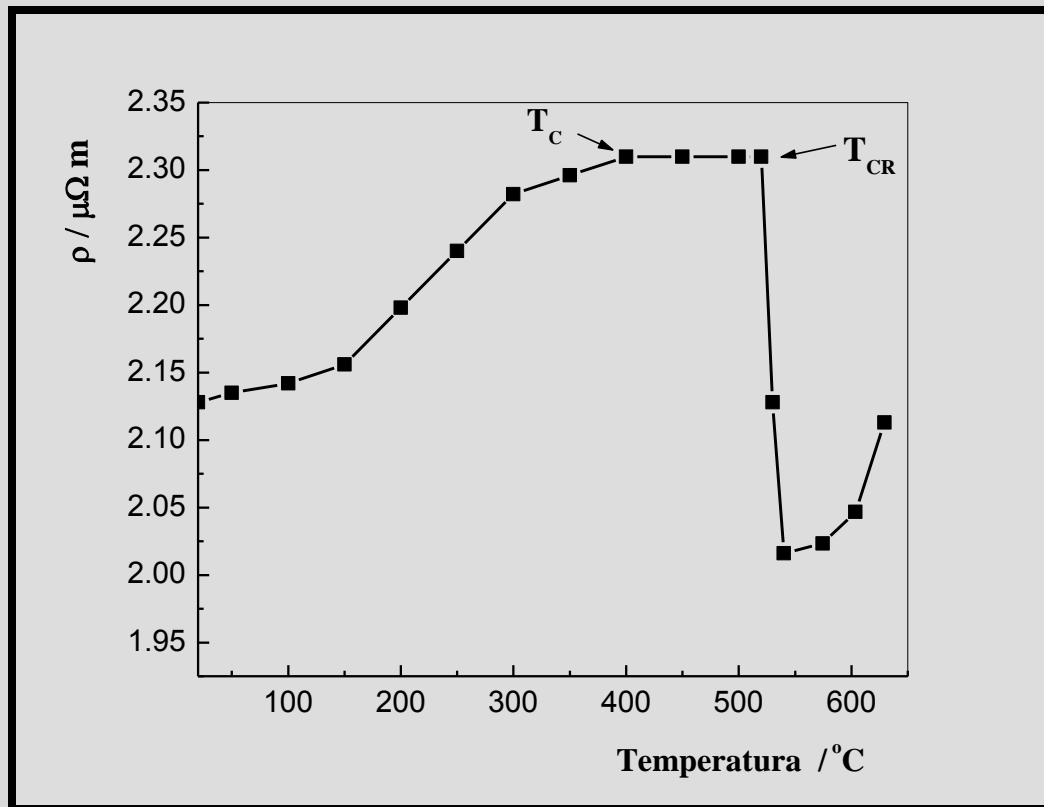
10/30/2009	HV	HFW	WD	mag	curr	3 μ m
12:37:11 PM	30.00 kV	13.5 μ m	30.1 mm	20000 x	30 pA	

FIB-slike poprečnog preseka
uzoraka odgrejanih na raznim
temperaturama tokom 1 časa



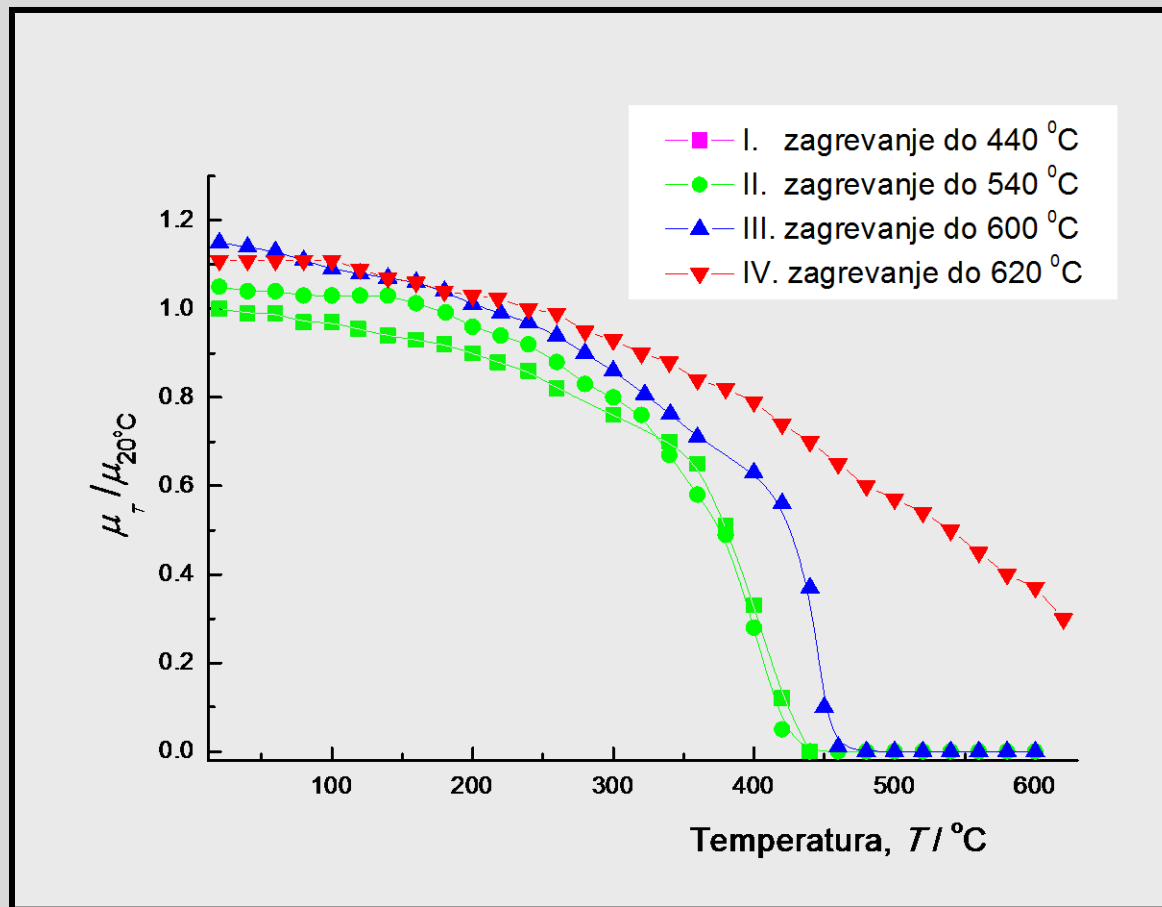
FIB-slika poprečnog preseka uzorka odgrevanog na 1123 tokom 24 časa

Uticaj strukturnih promena na električna svojstva



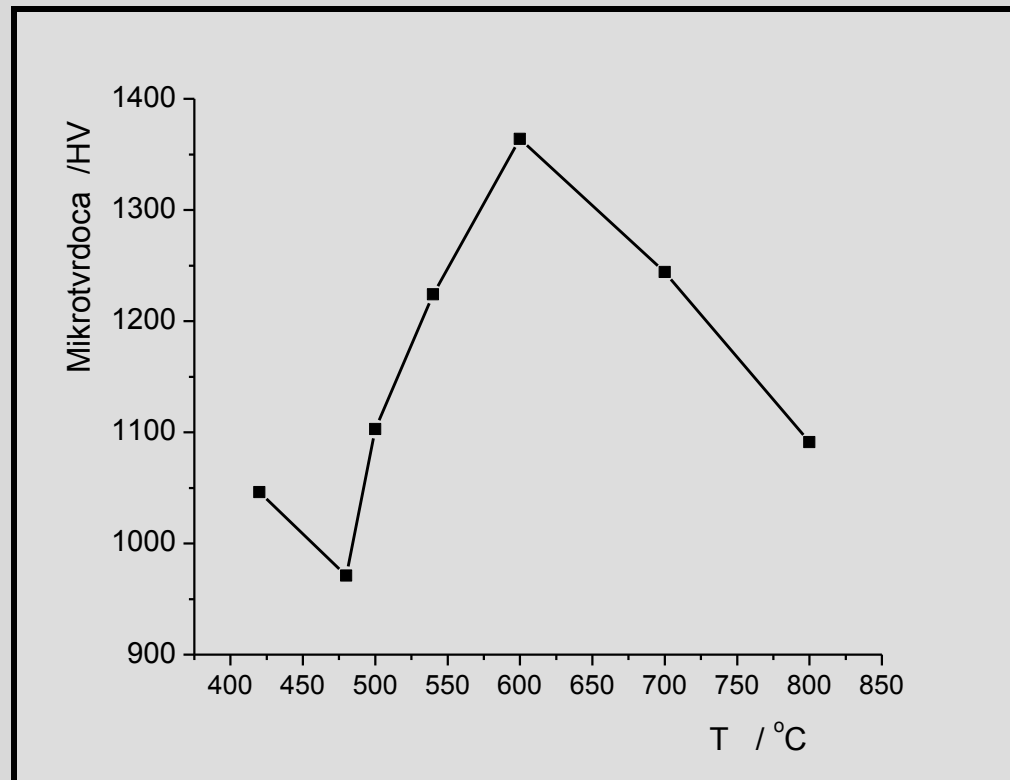
Temperaturska zavisnost
električne otpornosti
polazne legure.

Uticaj strukturnih promena na magnetna svojstva



Temperaturska zavisnost relativne magnetne susceptibilnosti tokom četiri uzastopna ciklusa zagrevanja legure do različitih temperatura.

Uticaj strukturnih promena na mikrotvrdoću



Temperaturska zavisnost mikrotvrdoće uzoraka legure odgrevanih na raznim temperaturama.

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