

SINTESIS, STRUKTUR KRISTAL DAN SIFAT MAGNETIK SUPERKONDUKTOR $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE=Nd, Eu, Gd)

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**Promotor : Prof. Dr. Suasmoro
Co-Promotor : Prof. Dr. Darminto
Co-Promotor : Suminar Pratapa, Ph.D.**

PENDAHULUAN

Latar Belakang

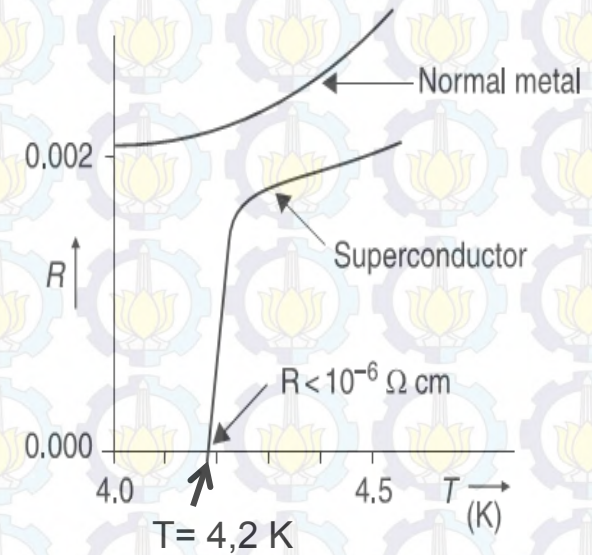
Aplikasi Superkonduktor



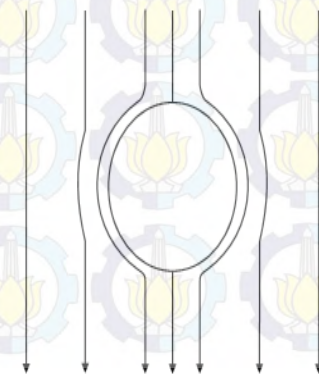
← Kabel transmisi daya



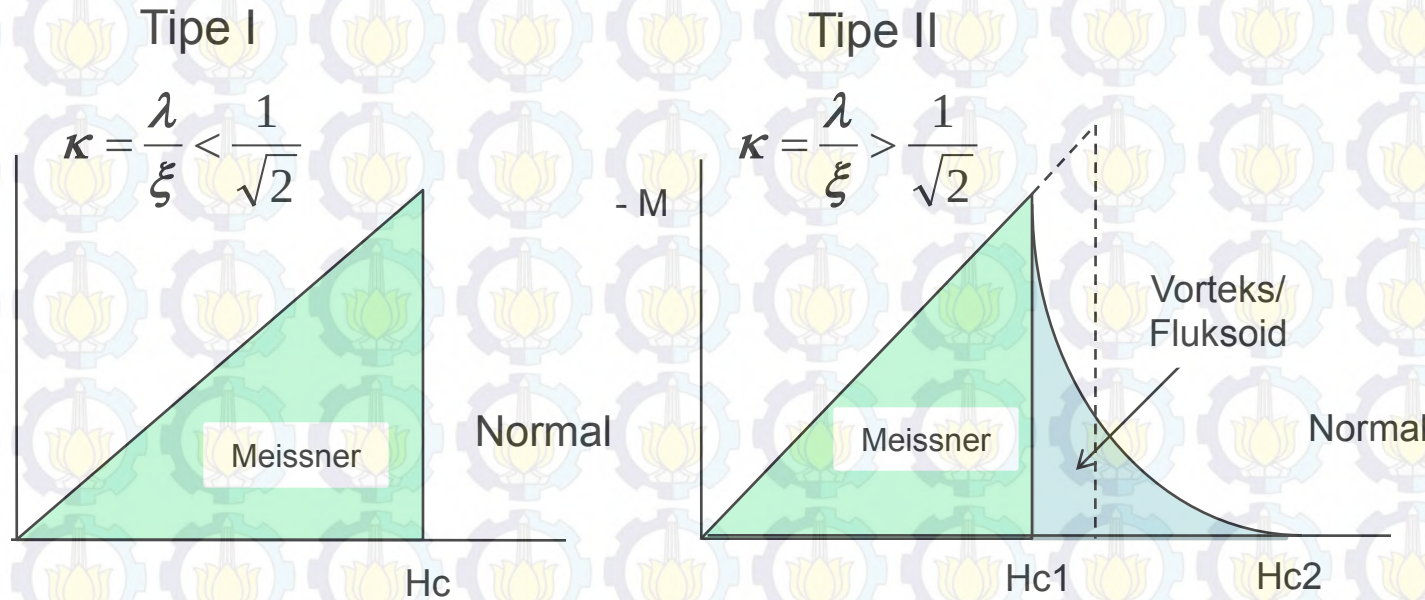
← MAGLEV



Efek Meissner



Tipe Superkonduktor



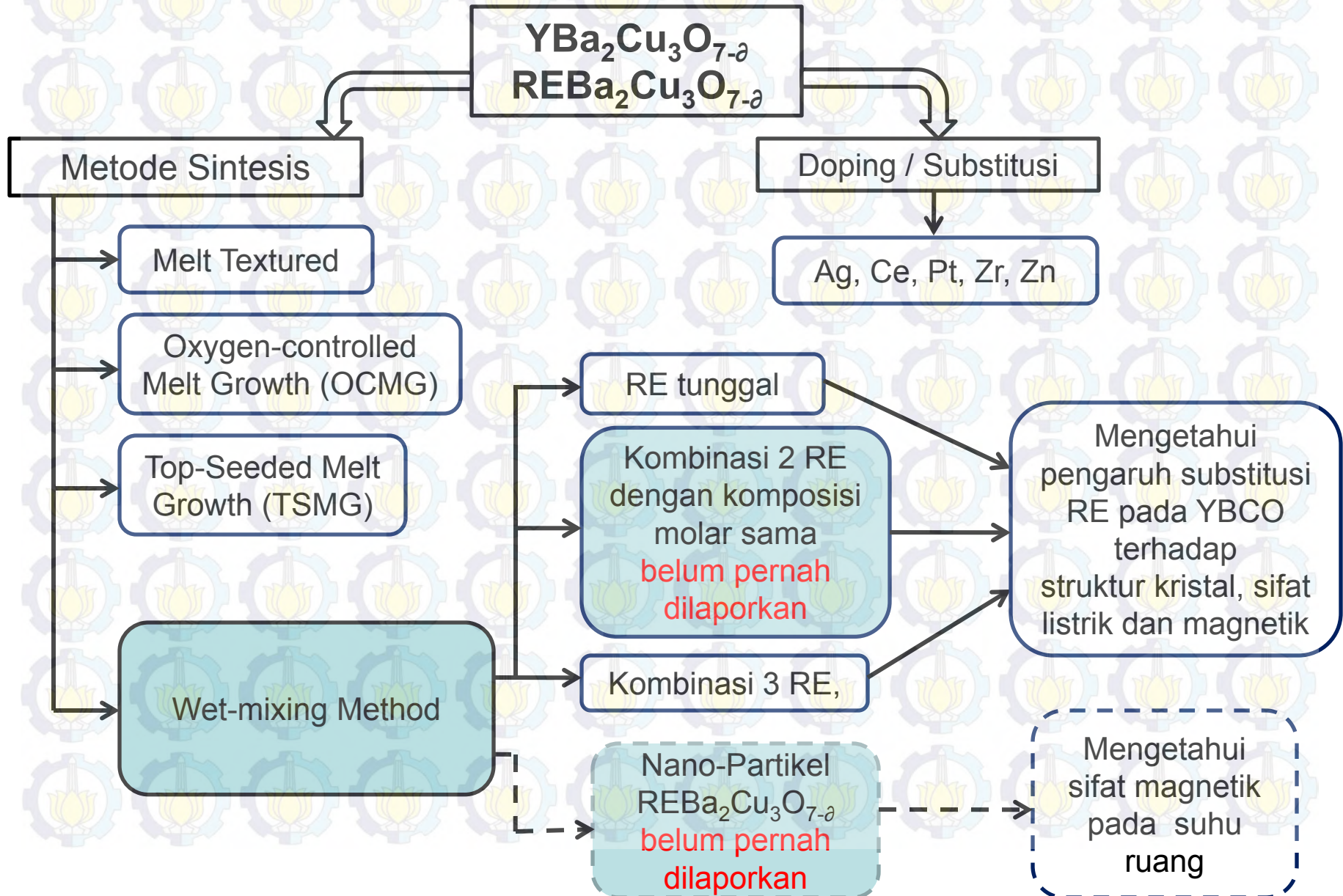
Tipe I

Logam		Campuran Logam	
Unsur	Tc (K)	Senyawa	Tc (K)
Al	1,2	V ₃ Ga	16,5
Hg	4,3	Nb ₃ Ge	23,2

Tipe II

Keramik Oksida		
Senyawa	Fasa	Tc (K)
Y-Ba-Cu-O	Y-123	90,0
Bi-Sr-Ca-Cu-O	Bi-2101	30,0
	Bi-2212	80,0
	Bi-2223	110,0

Latar Belakang



SINTESIS SUPERKODUKTOR $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$

Proses Sintesis $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$

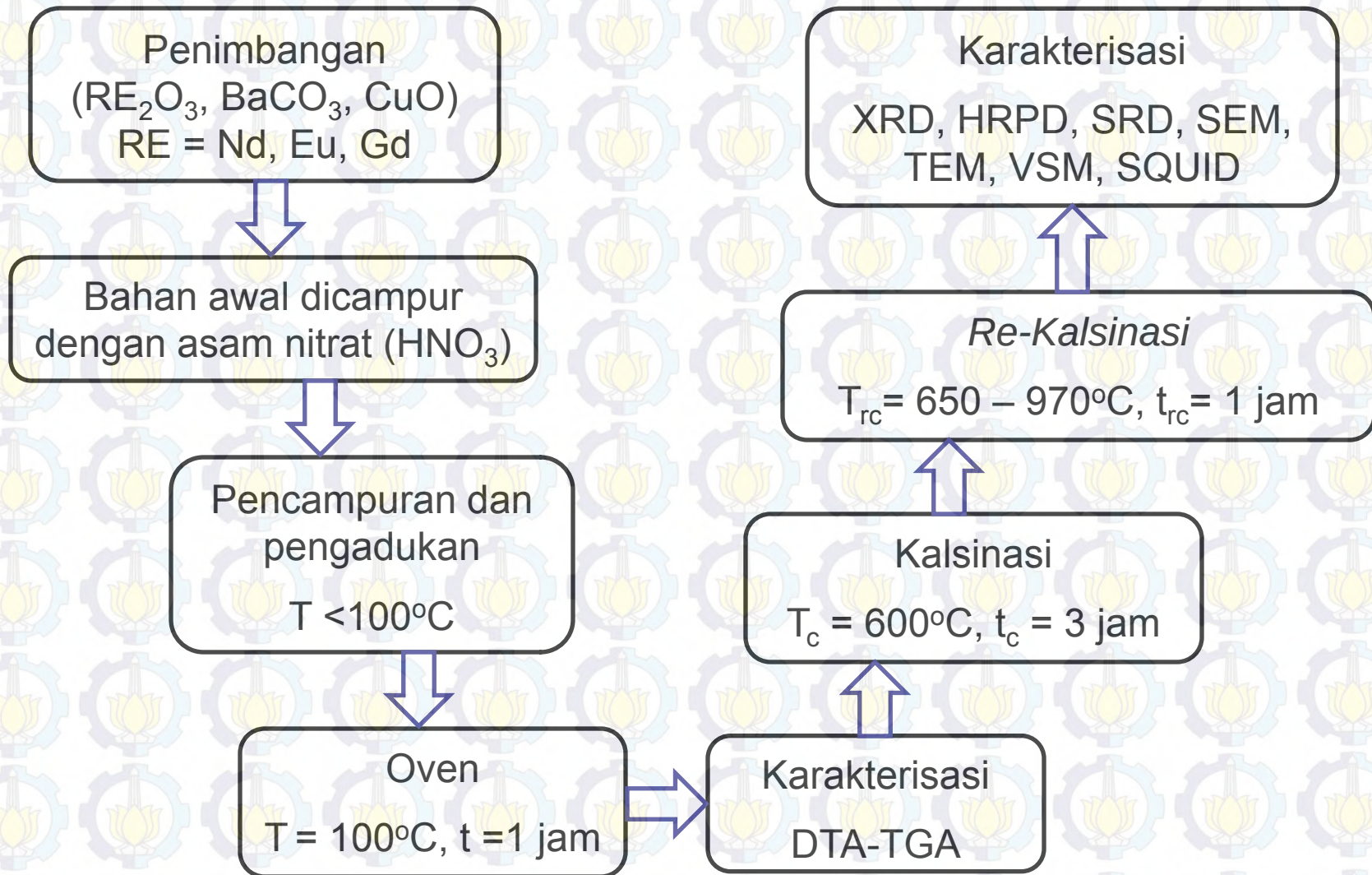
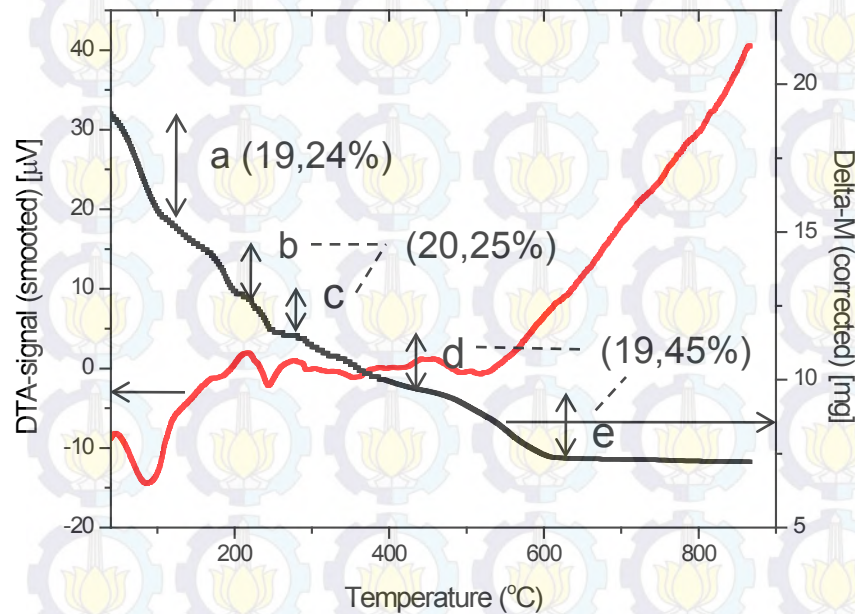
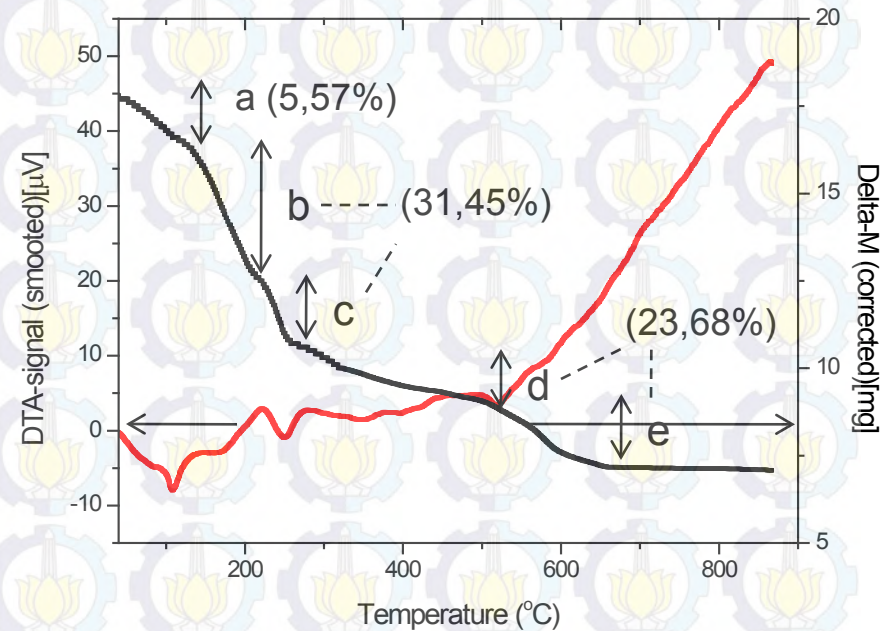


Diagram alir sintesis superkonduktor $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ dengan wet-mixing method



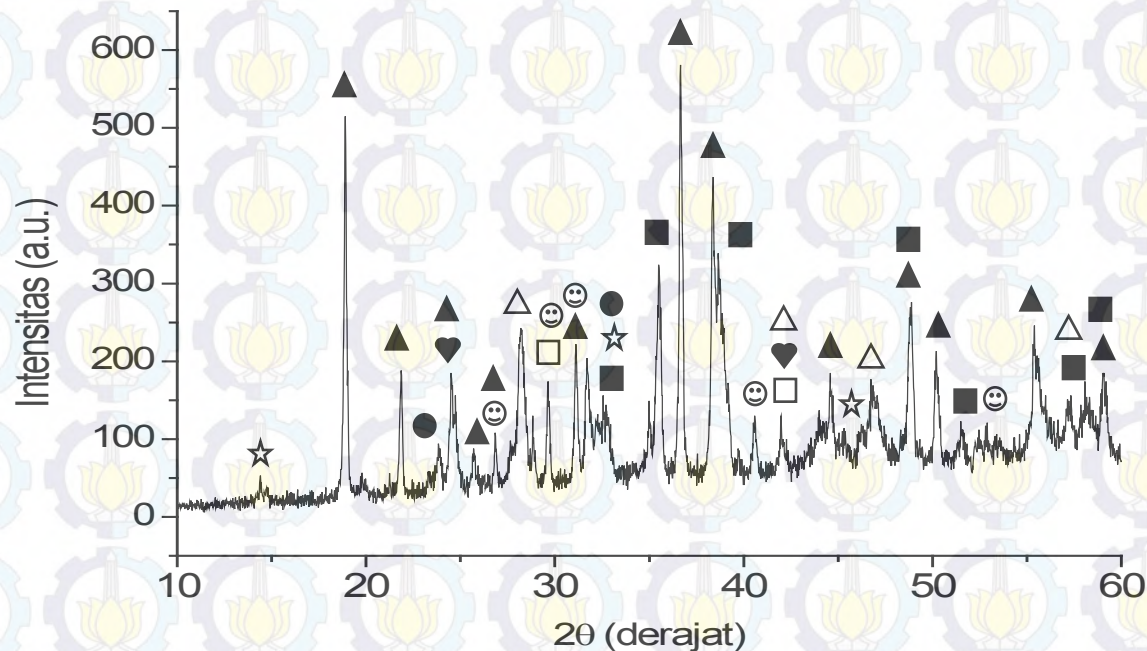
Hasil karakterisasi DTA-TGA sampel $NdBa_2Cu_3O_{7-\delta}$



Hasil karakterisasi DTA-TGA sampel $GdBa_2Cu_3O_{7-\delta}$

Identifikasi Fasa

Pola difraksi sinar-X sampel $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$
($T_{\text{cal}} = 600^\circ\text{C}$, $t_{\text{cal}} = 3 \text{ jam}$)



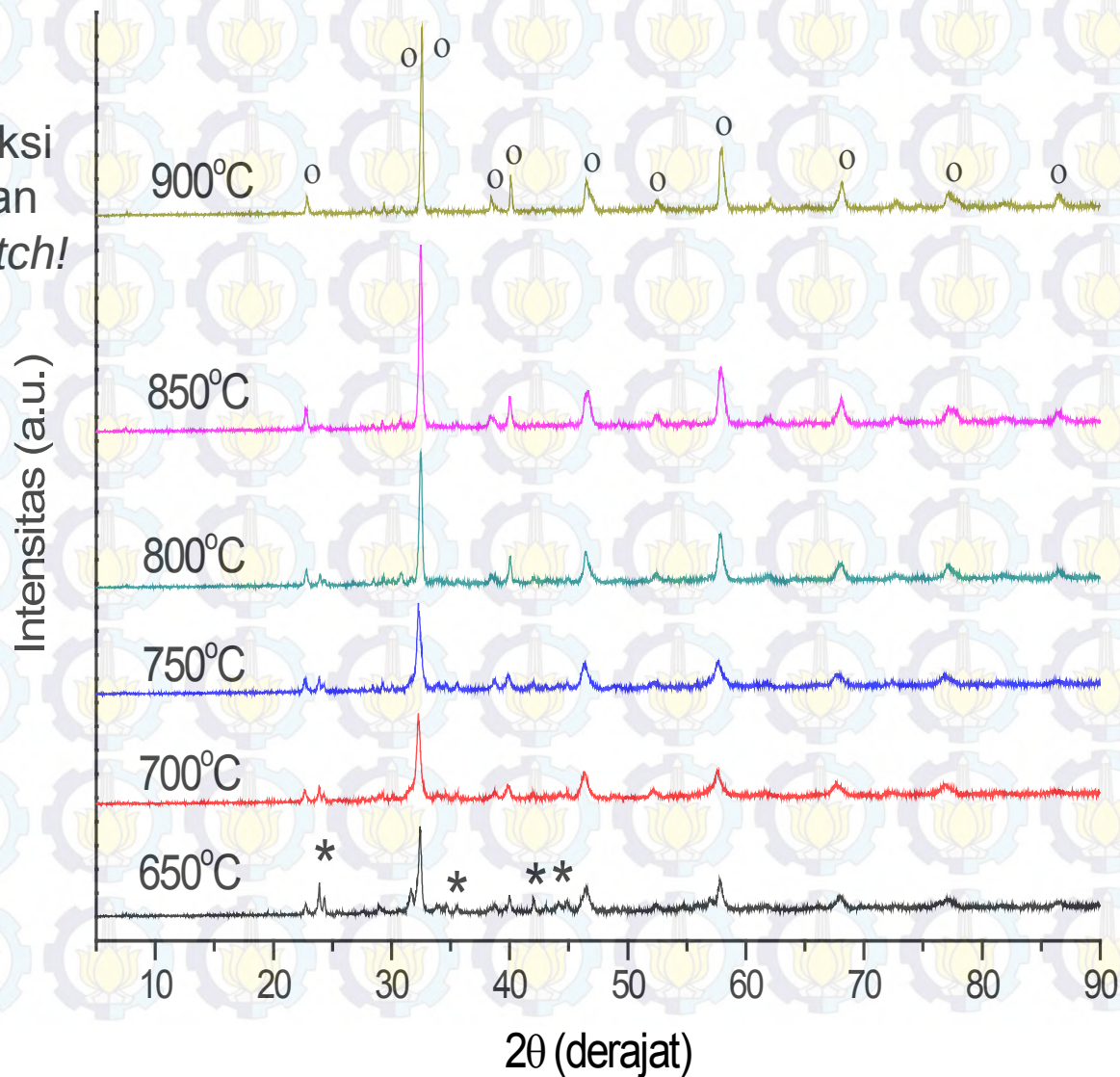
Fasa – fasa yang terbentuk :

- ▲ $\text{Ba}(\text{NO}_3)_2$, ■ CuO , ● Nd_2CuO_4 ,
- ♥ BaCO_3 , □ BaNd_2O_4 , △ BaCuO_2 ,
- ☺ Nd_2O_3 , ☆ $(\text{Nd}_{0.925}\text{Ba}_{0.075-x})_2\text{CuO}_{4-x}$

Pola Difraksi Sinar-X Nd-123

Pola difraksi sinar-X sampel $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$
($T_{\text{rc}} = 650\text{-}900^\circ\text{C}$, $t_{\text{rc}} = 1$ jam)

Pencocokan
puncak difraksi
menggunakan
program *Match!*



Fraksi Berat Nd-123

Fraksi berat sampel Nd-123
($T_{rc} = 650-900^{\circ}\text{C}$, $t_{rc} = 1 \text{ jam}$)

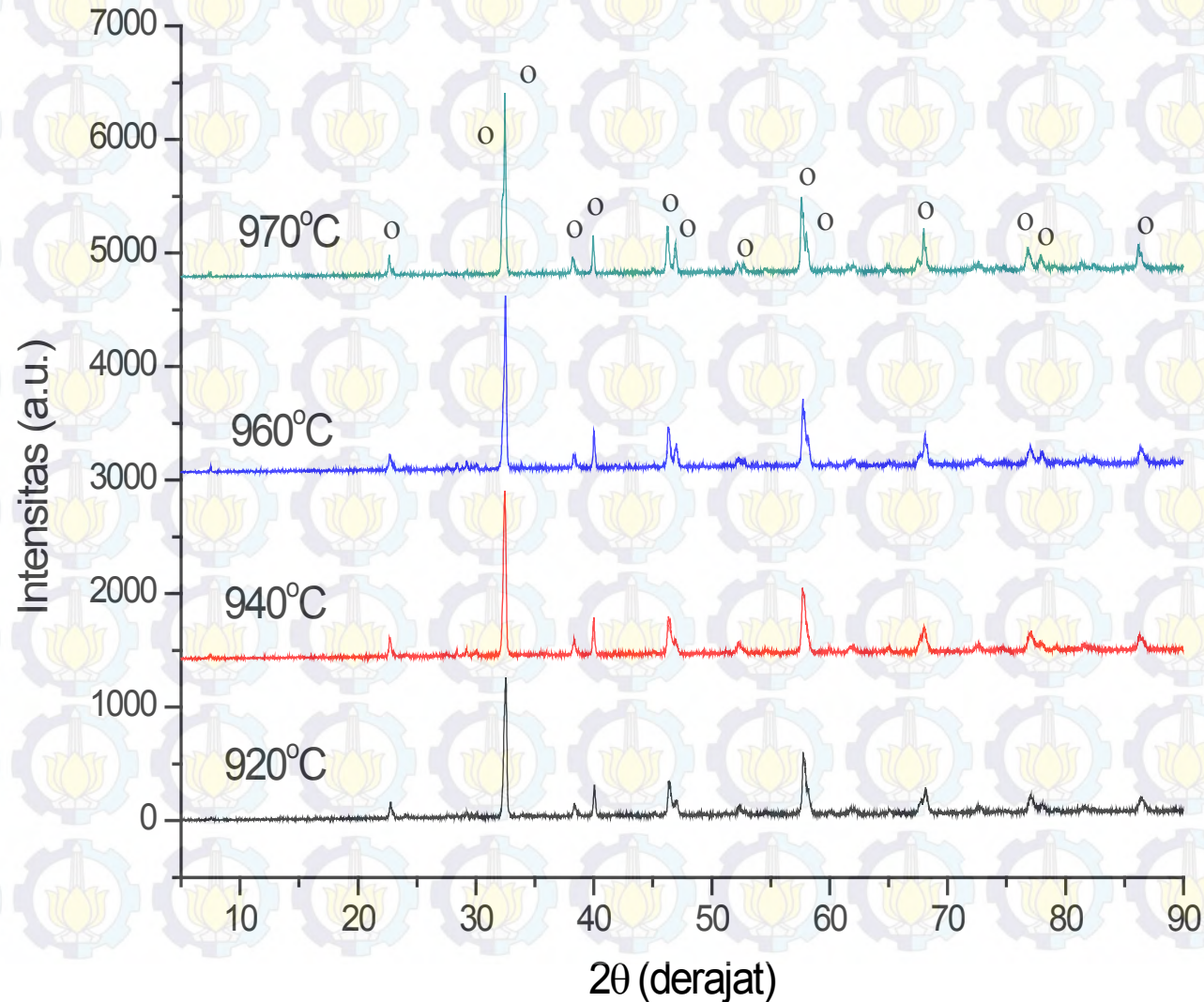
Fraksi berat (FB) dihitung menggunakan program Rietica

$T_{rc} (^{\circ}\text{C})$	Fraksi Berat (%)						
	Nd-123	BaCO_3	BaCuO_2	CuO	Nd_2CuO_4	$\text{Nd}_2\text{BaCuO}_5$	BaNd_2O_4
650	84,3	13,2	0,4	1,7	0,4	-	-
700	86,2	10,4	0,9	1,5	0,2	-	0,8
750	87,6	8,4	1,6	1,2	0,1	-	1,1
800	89,2	5,9	2,4	0,7	-	0,6	1,2
850	90,8	4,2	3,1	0,3	-	0,5	1,1
900	92,4	2,0	4,2	0,1	-	0,2	1,1

Semakin tinggi suhu re-kalsinasi,
fraksi berat Nd-123 semakin besar

Pola Difraksi Sinar-X Nd-123

Pola difraksi sinar-X sampel $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$
($T_{\text{rc}} = 920\text{-}970^\circ\text{C}$, $t_{\text{rc}} = 1$ jam)



Fraksi Berat Nd-123

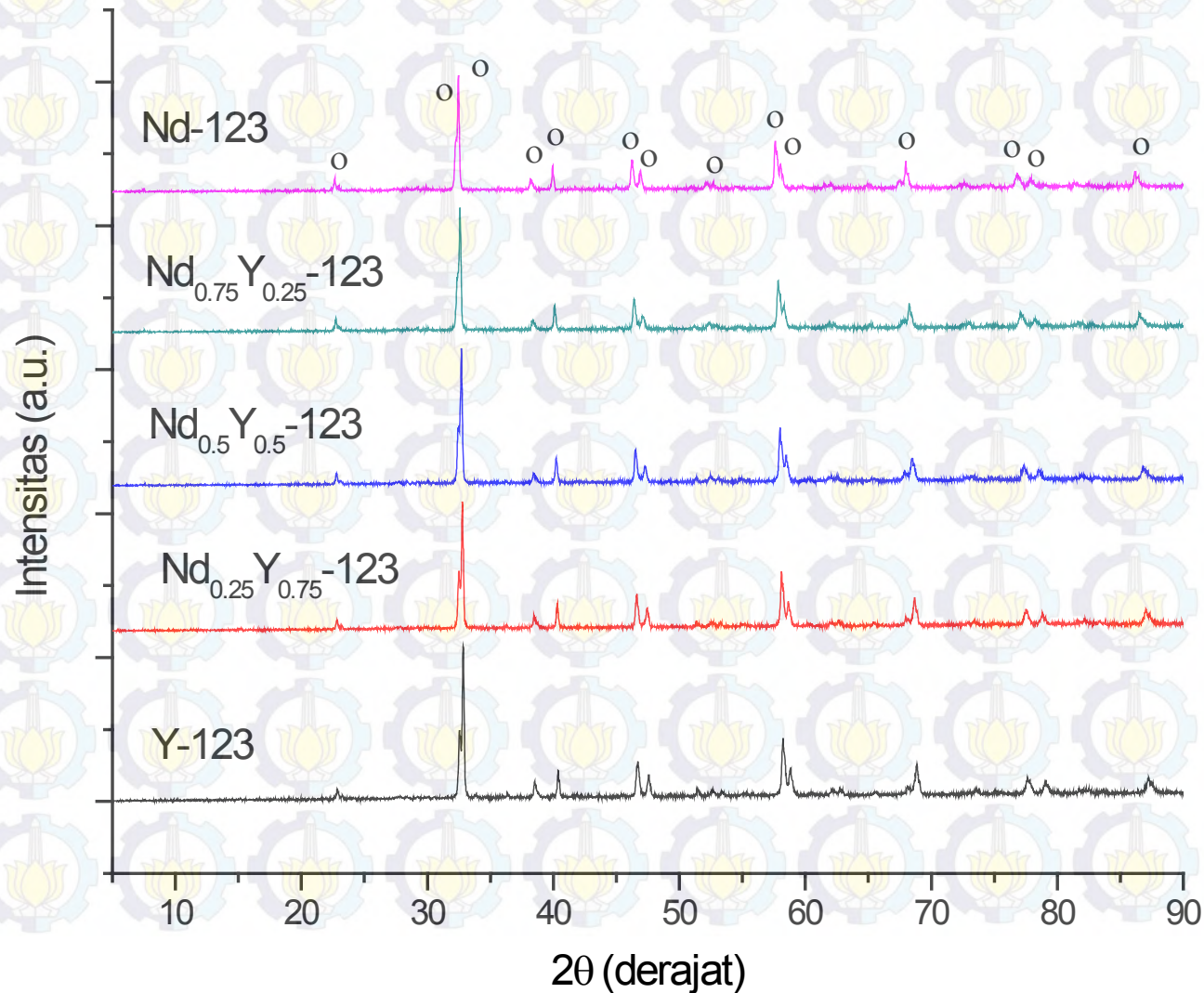
Fraksi berat sampel Nd-123
($T_{rc} = 920-970^{\circ}\text{C}$, $t_{rc} = 1$ jam)

$T_{rc} (^{\circ}\text{C})$	Fraksi Berat (%)		
	Nd-123	BaCuO ₂	BaNd ₂ O ₄
920	94,2	4,1	1,7
940	94,7	3,8	1,5
960	95,2	3,1	1,7
970	95,9	2,5	1,6

$T_{rc} = 970^{\circ}\text{C}$ digunakan sebagai
acuan untuk sintesis sampel
berikutnya

Pola Difraksi Sinar-X NY-123

Pola difraksi sinar-X sampel $\text{Nd}_x\text{Y}_{(1-x)}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x=0-1$)
($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)



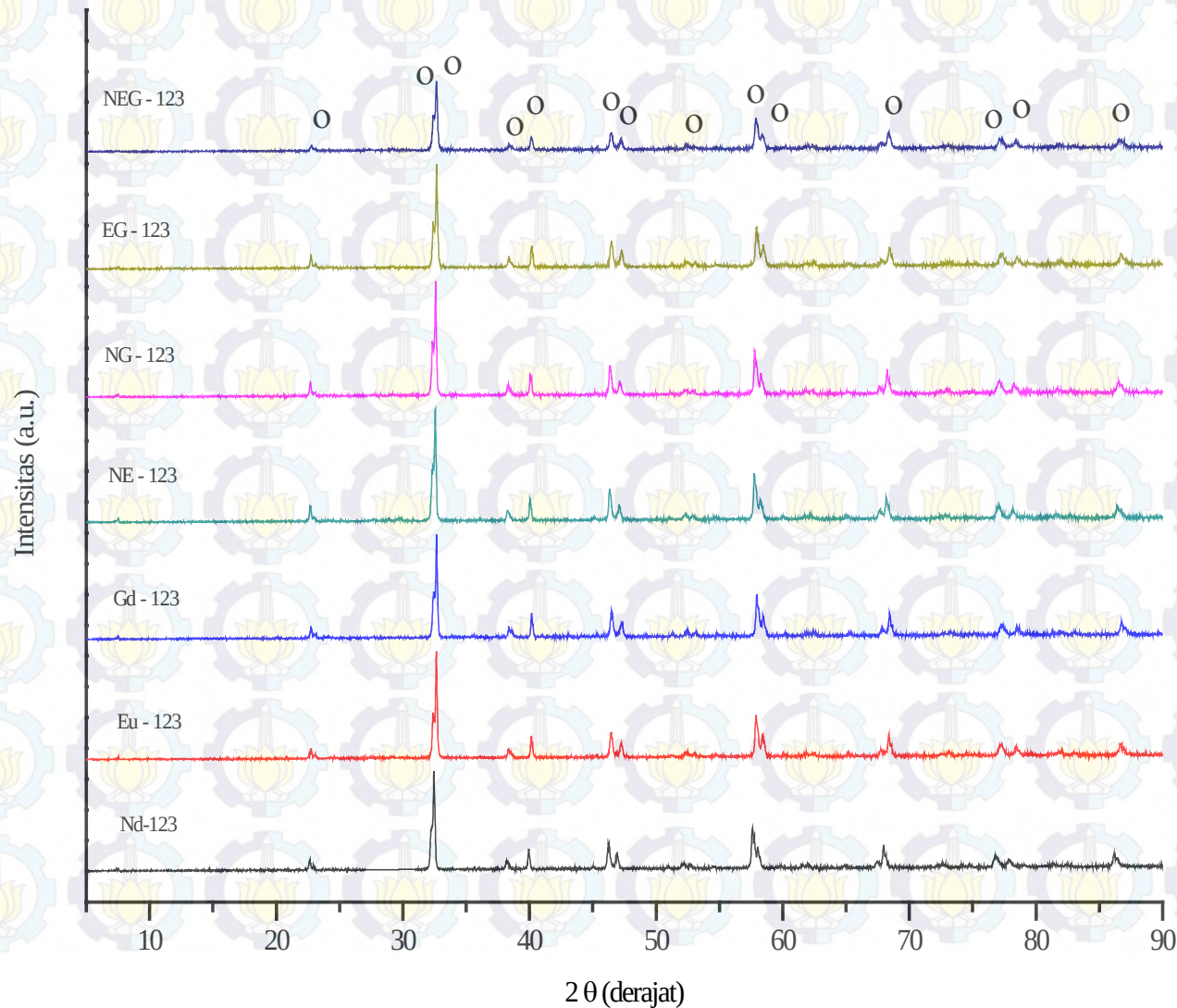
Fraksi Berat NY-123

Fraksi berat sampel NY-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

Sampel	Fraksi Berat (%)		
	NY-123	BaCuO ₂	BaNd ₂ O ₄ / BaY ₂ O ₄
Nd-123	97,8	1,7	0,5
Nd _{0.75} Y _{0.25} -123	96,7	2,1	1,2
Nd _{0.5} Y _{0.5} -123	96,8	1,9	1,3
Nd _{0.25} Y _{0.75} -123	97,0	2,2	0,8
Y-123	96,6	2,1	1,3

Pola difraksi sinar-X sampel RE-123

Pola difraksi sinar-X sampel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE=Nd, Eu, Gd)
($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)



Fraksi Berat RE-123

Fraksi berat sampel RE-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

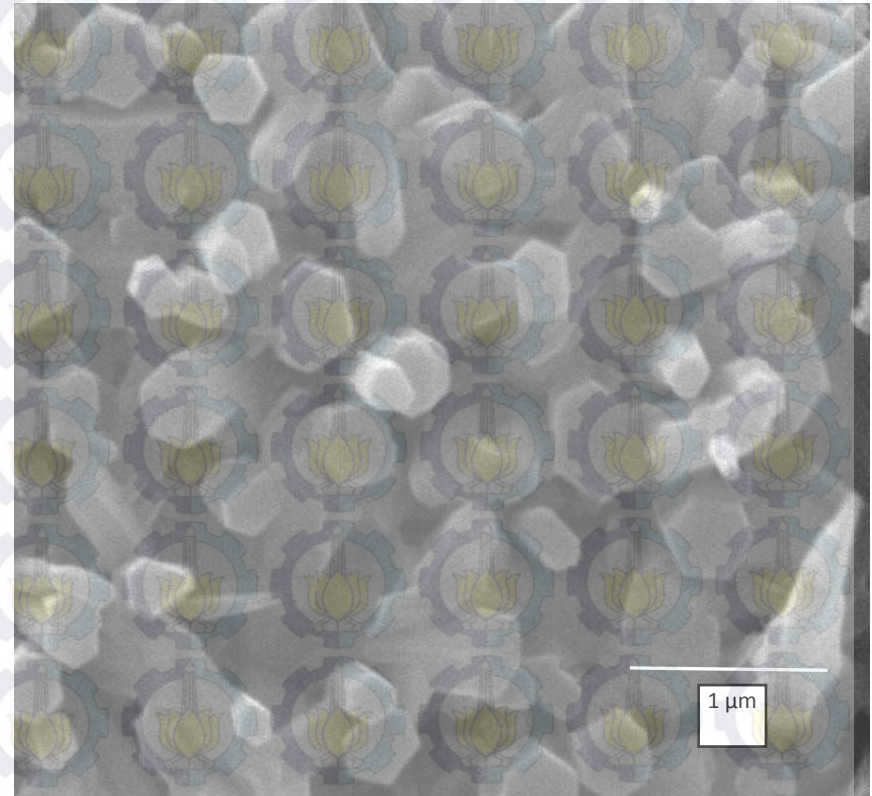
Sampel	Fraksi Berat (%)		
	RE-123	BaCuO ₂	BaRE ₂ O ₄
Nd-123	97,8	1,7	0,5
Eu-123	98,4	0,9	0,7
Gd-123	99,1	0,7	0,2
NE-123	97,5	1,8	0,7
NG-123	98,0	1,2	0,8
EG-123	98,8	0,9	0,3
NEG-123	98,1	1,4	0,5

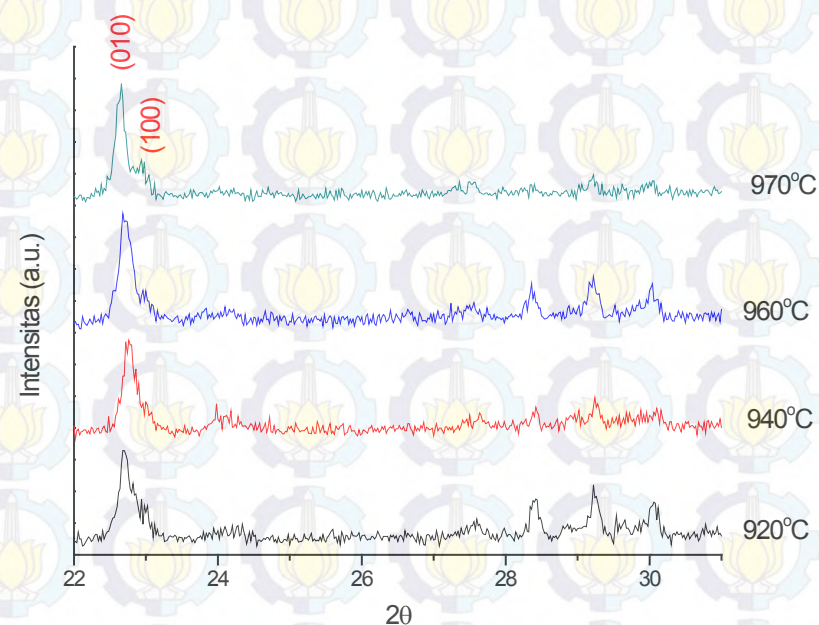
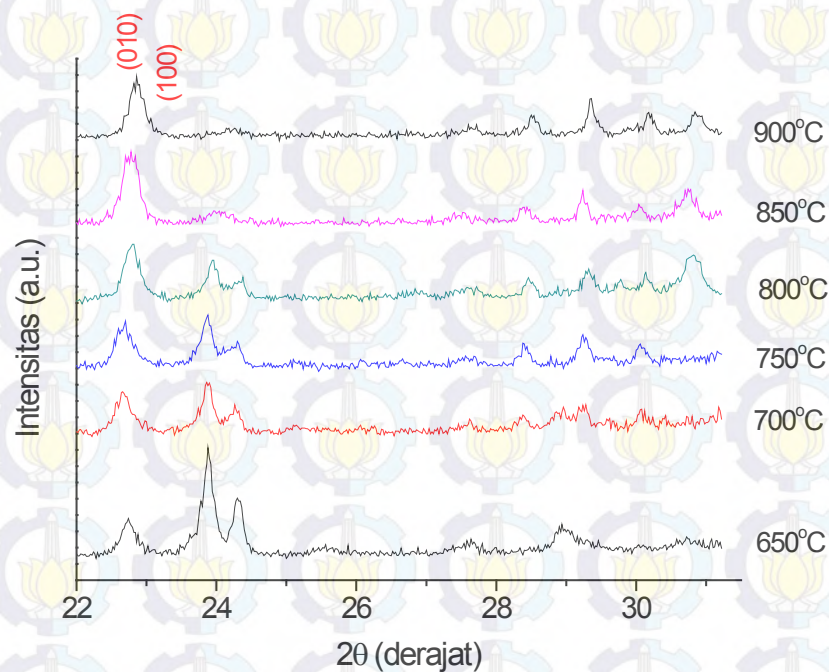
Morfologi (TEM, SEM) Nd-123

Gambar TEM sampel Nd-123
($T_{rc} = 700^{\circ}\text{C}$, $t_{rc} = 1 \text{ jam}$)



Gambar SEM sampel Nd-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 1 \text{ jam}$)





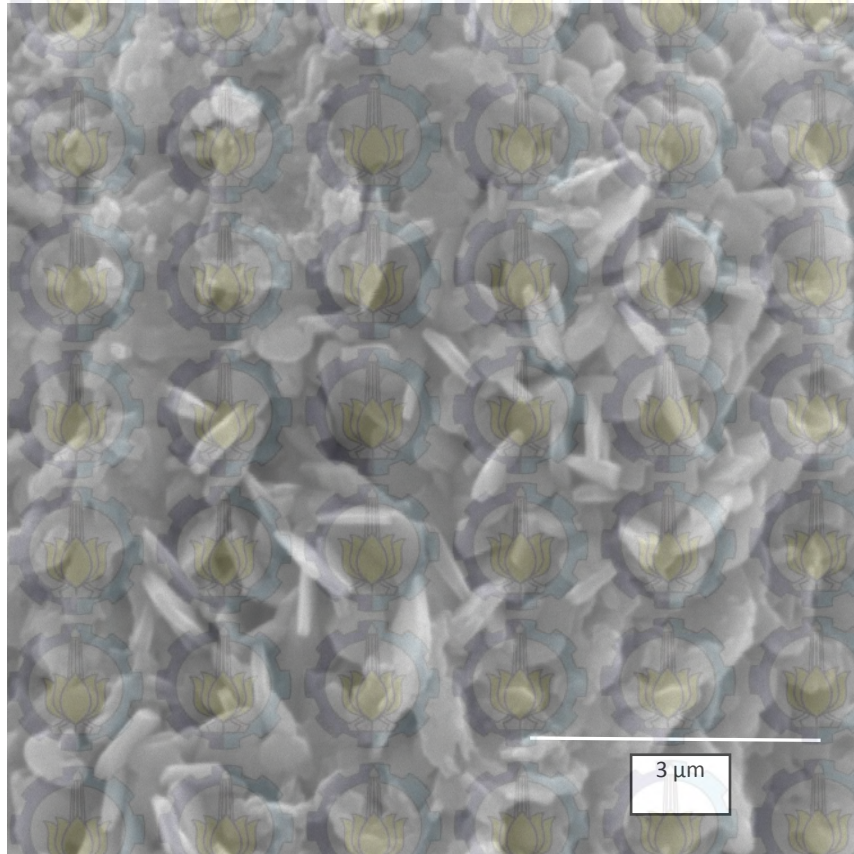
PDF No. 00-046-0229

No	2θ (derajat)	d (Å)	(hkl)
1	22,72	3,91	(010)
2	22,91	3,88	(100)

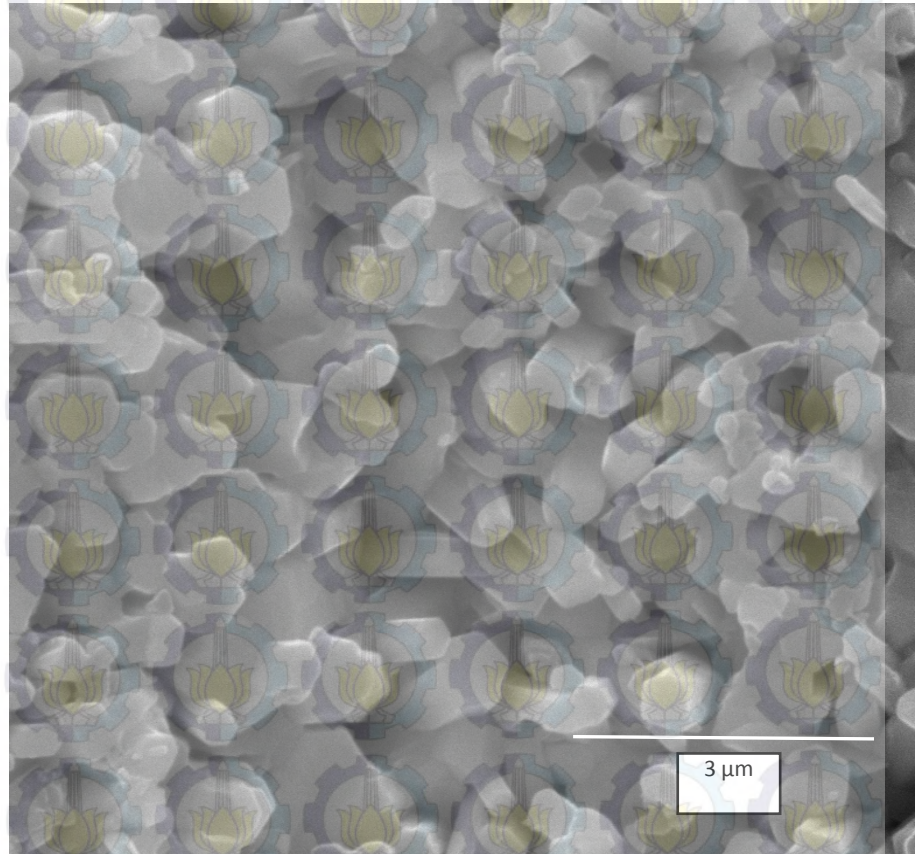
Antipov, E., Abakumov, A., Moscow State Univ., Russia, ICDD Grant-in-Aid, (1994)

Morfologi (SEM) NY-123

Gambar SEM sampel NY-123 ($x=0$)
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)



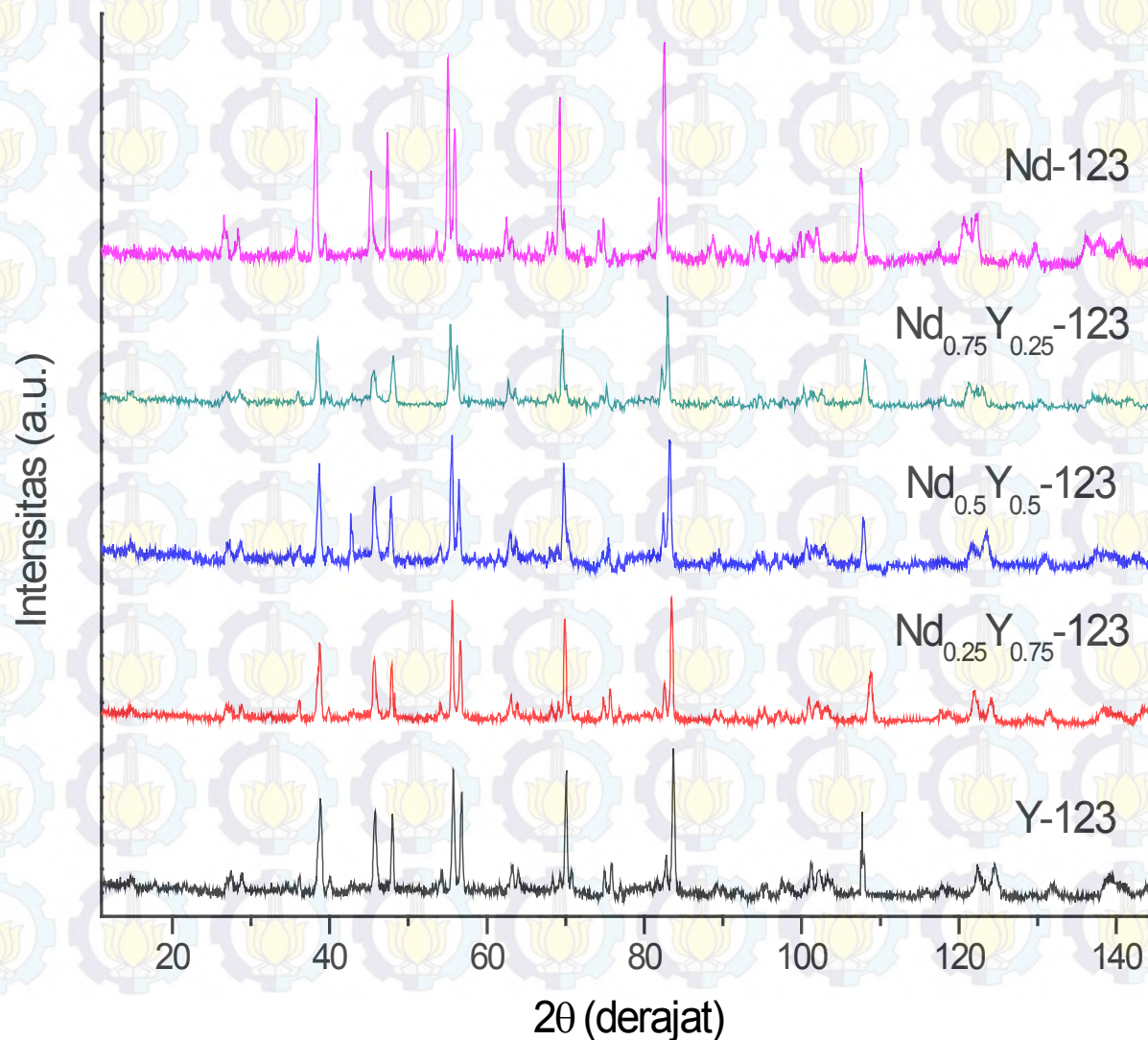
Gambar SEM sampel NY-123 ($x=1$)
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)



STRUKTUR KRISTAL

Pola difraksi netrom (HRPD)

Pola difraksi netron (HRPD) sampel $\text{Nd}_x\text{Y}_{(1-x)}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x=0-1$)
($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)

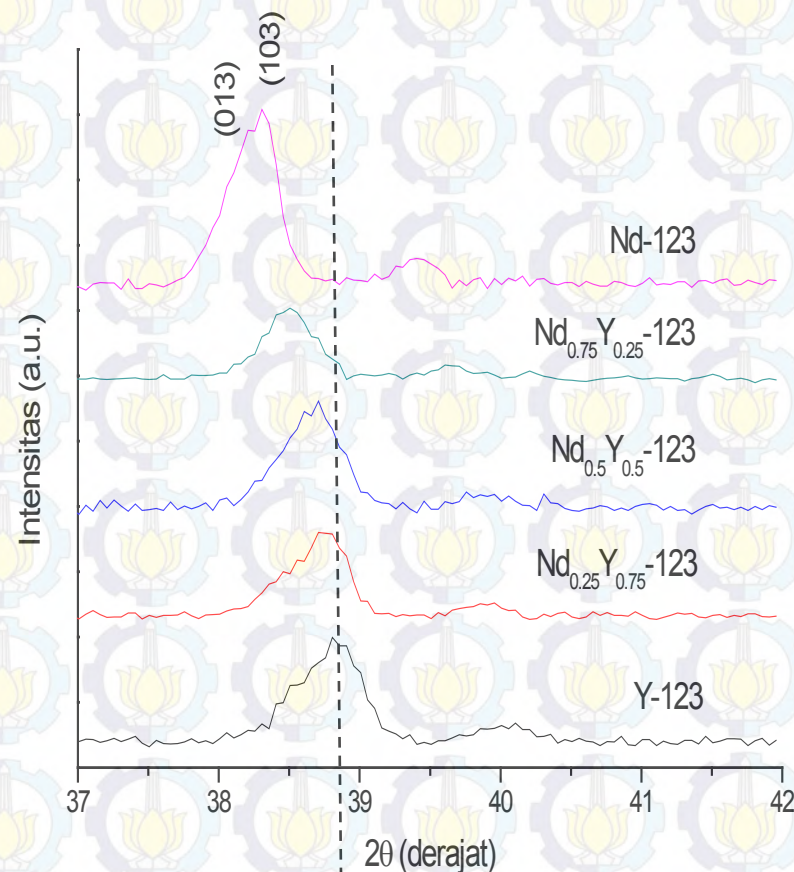
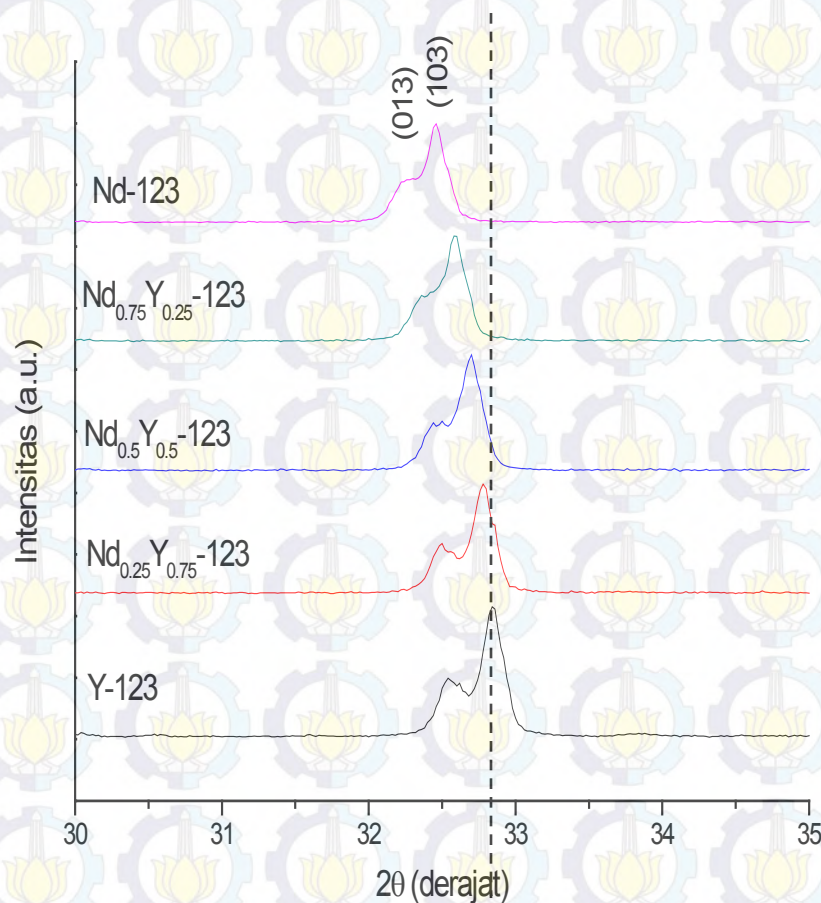


HRPD dilakukan di
PTBIN BATAN
Serpong

Pergeseran sudut

XRD Sampel $\text{Nd}_x\text{Y}_{(1-x)}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$
($x=0-1$) ($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)
Pada sudut $30-35^\circ$

HRPD Sampel $\text{Nd}_x\text{Y}_{(1-x)}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$
($x=0-1$) ($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)
pada sudut $37-42^\circ$



Pergeseran sudut disebabkan oleh perbedaan jari-jari ion Nd^{3+} dan Y^{3+}
 $\text{Y}^{3+} (104 \text{ pm}) < \text{Nd}^{3+} (112.3 \text{ pm})$

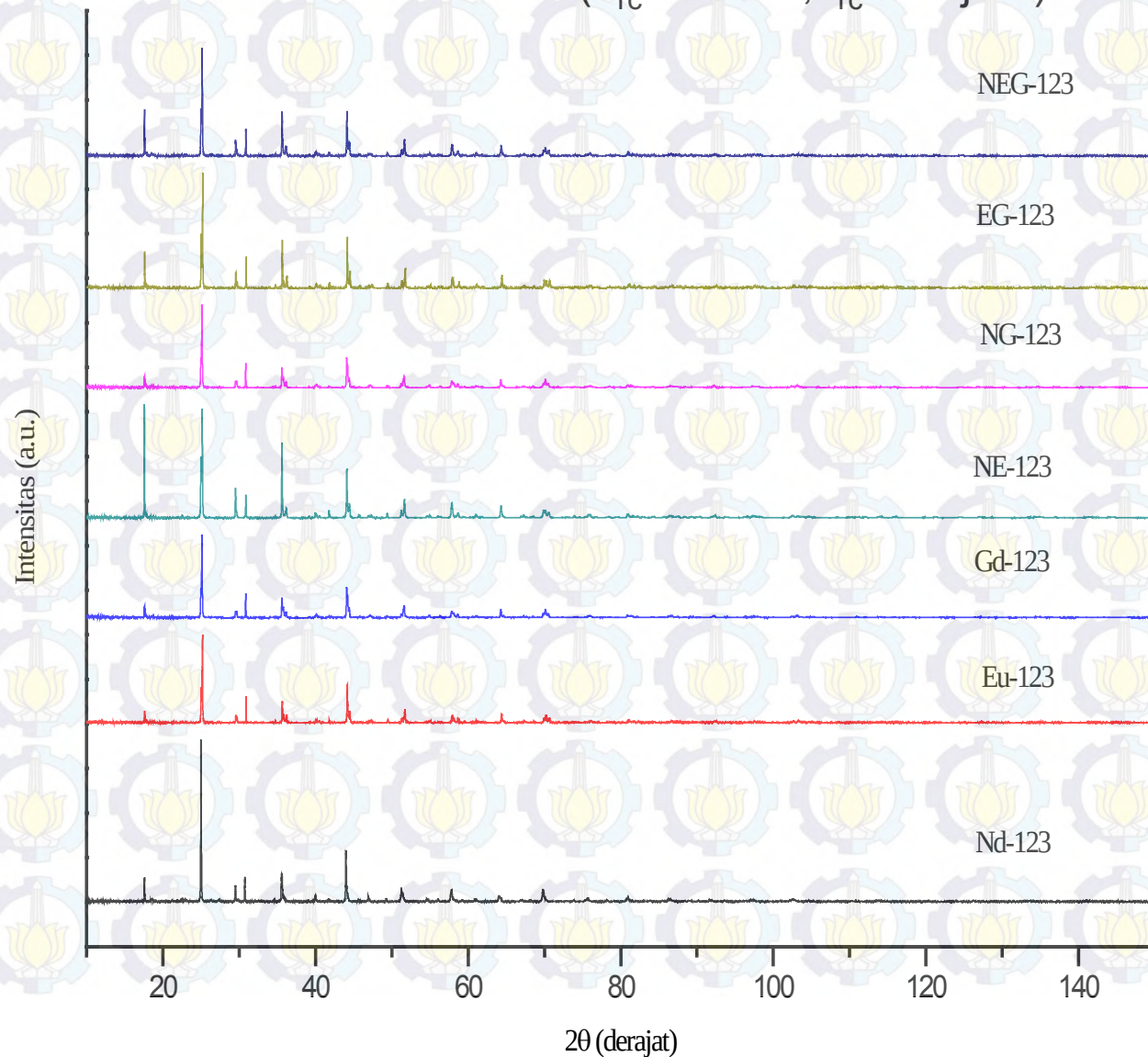
Parameter kisi (a, b, c) berubah

Ortorombik :
$$\frac{1}{d_{hkl}} = \sqrt{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}}$$

Bragg :
$$n\lambda = 2d \sin \theta$$

Pola difraksi sinkrotron (SRD)

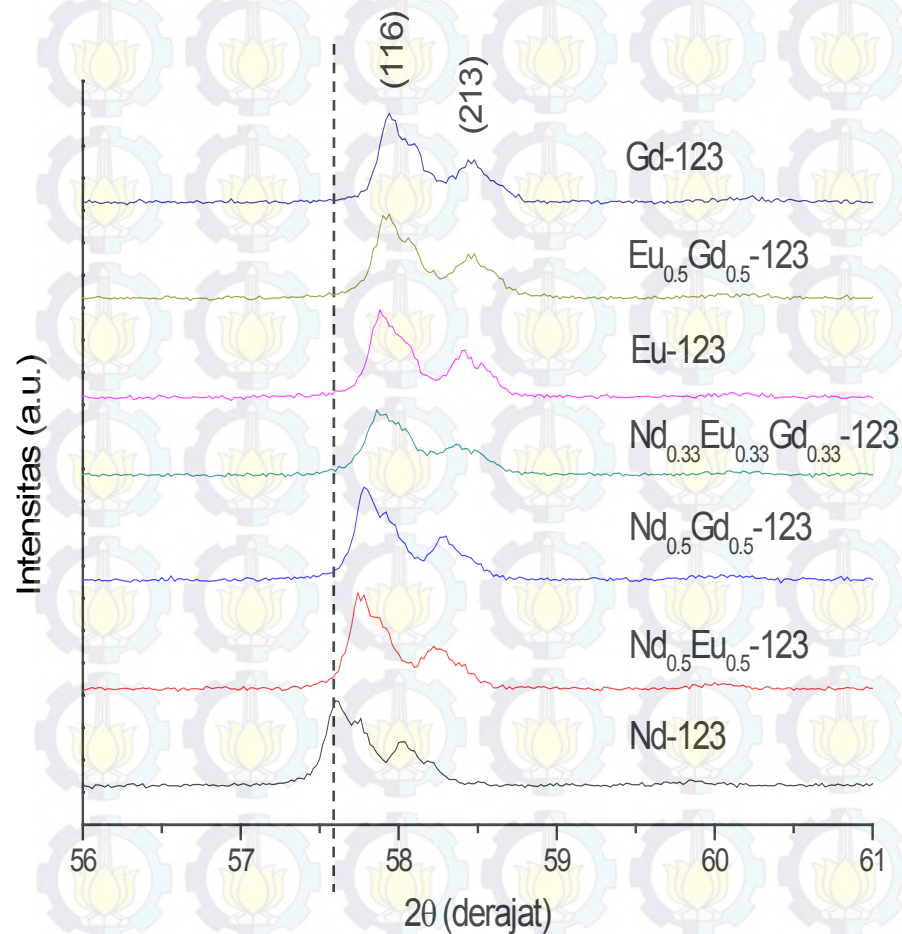
Pola difraksi sinkrotron Sampel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE=Nd, Eu, Gd)
($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)



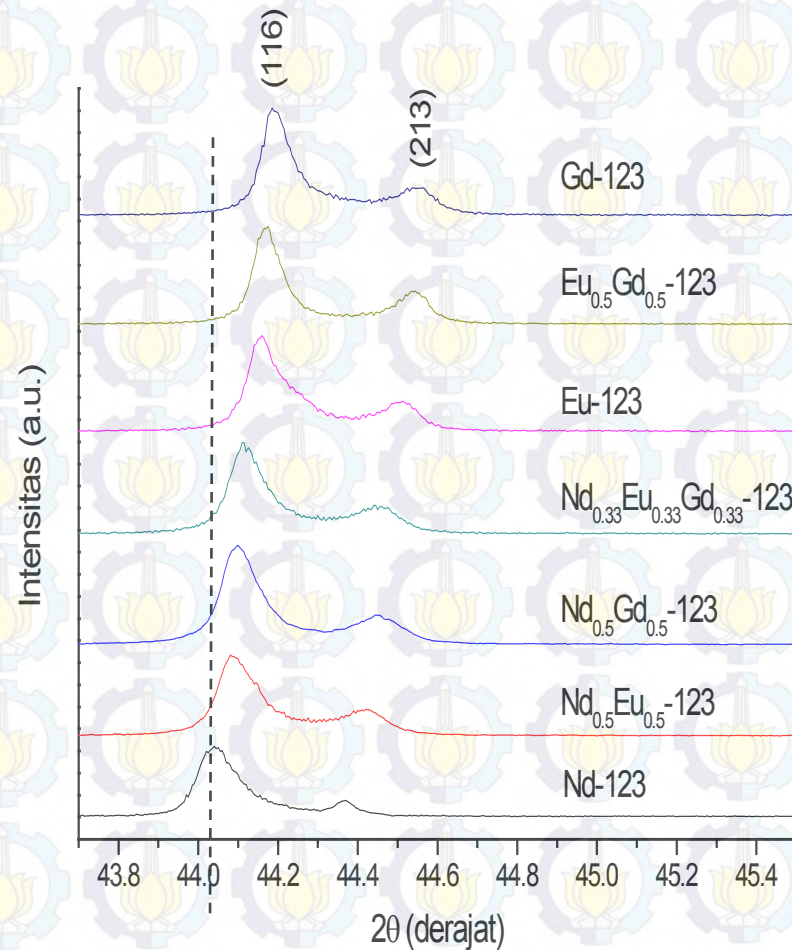
SRD dilakukan di
KEK (The High
Energy Accelerator
Research
Organization)
Jepang

Pergeseran sudut

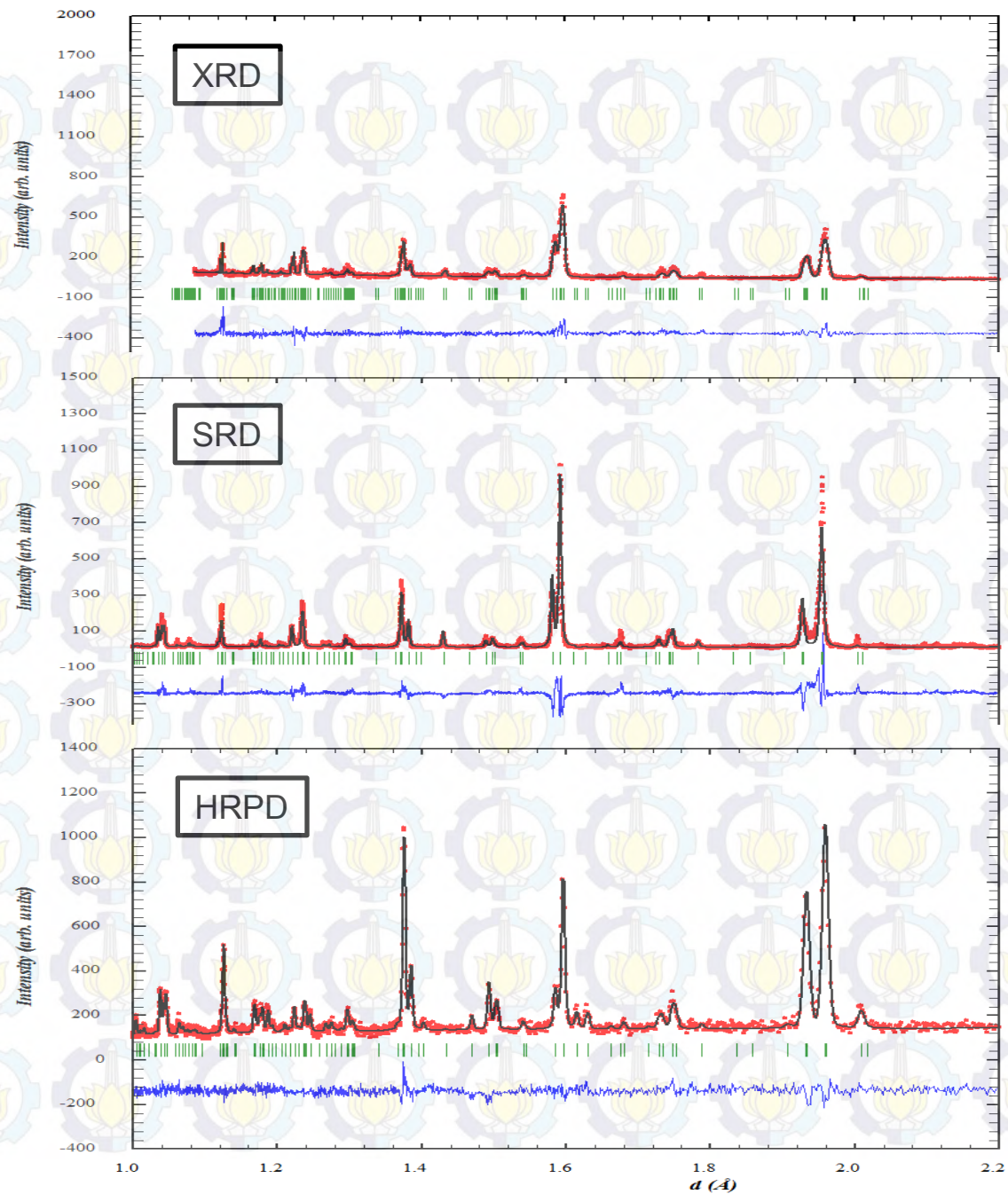
XRD sampel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$
(RE=Nd, Eu, Gd) ($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)
pada sudut $56\text{--}61^\circ$



SRD sampel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$
(RE=Nd, Eu, Gd) ($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)
pada sudut $43,8\text{--}45,5^\circ$



Hasil refinement *XRD, SRD* *dan HRPD*



Hasil Refinement XRD, SRD, HRPD

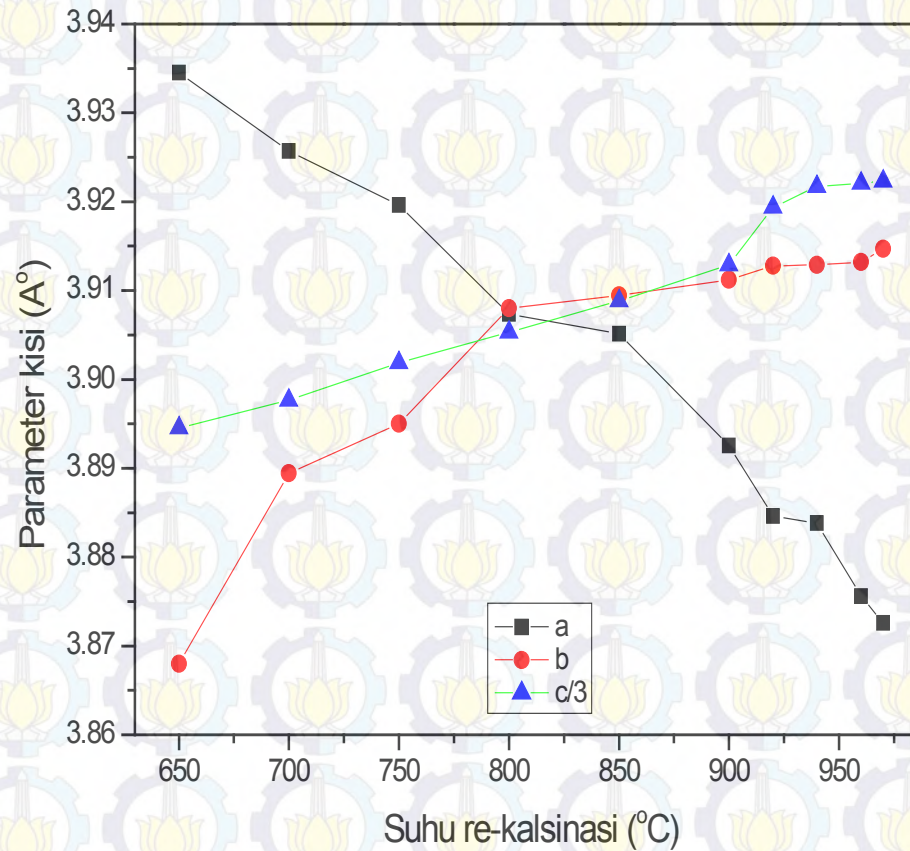
Harga parameter kisi a, b dan c, faktor *releability* (R_p , R_{wp} , R_e , R_B , R_F) dan χ^2 sampel Nd-123 hasil karakterisasi XRD, SRD dan HRPD

--	Nd-123 (XRD)	Nd-123 (SRD)	Nd-123 (HRPD)
a (Å)	3,872(6)	3,9008(5)	3,868(9)
b (Å)	3,922(1)	3,9113(6)	3,920(3)
c (Å)	11,775(4)	11,74(8)	11,767(9)
χ^2	1,1	1,2	1,3
R_p (%)	9,4	16,6	6,9
R_{wp} (%)	12,3	22,5	8,7
R_e (%)	11,6	20,3	7,7
R_B (%)	11,2	15,7	11,8
R_F (%)	11,3	9,6	9,1

Nama	PDF No.	Senyawa	a (Å)	b (Å)	c (Å)
Antipov, et.al	00-046-0229	NdBa ₂ Cu ₃ O _{6.88}	3,8799	3,9161	11,7645

Parameter Kisi Nd-123

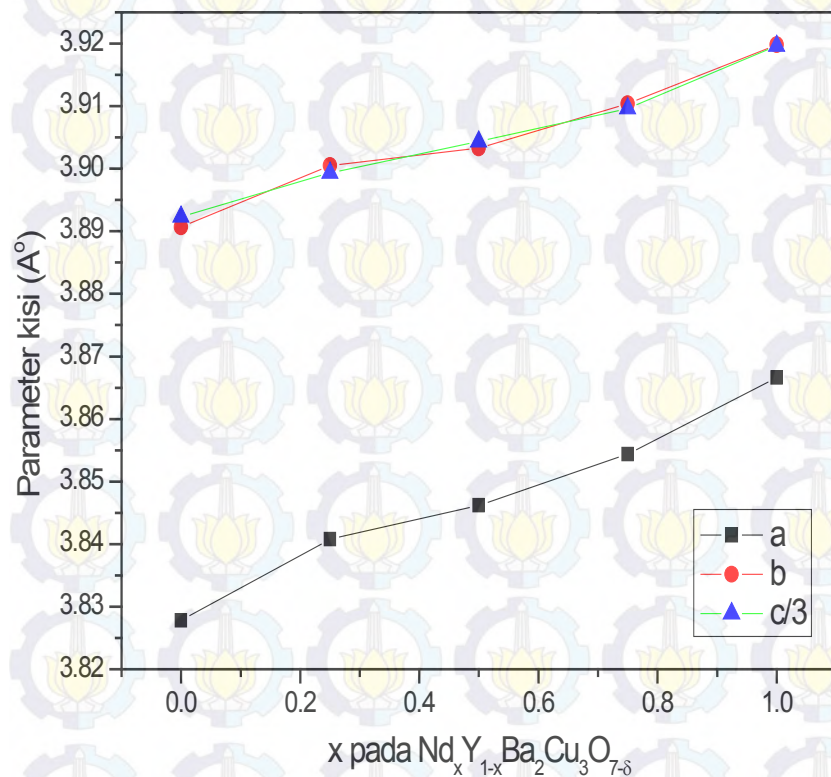
Parameter kisi sampel Nd-123
($T_{rc} = 650-970^{\circ}\text{C}$, $t_{rc} = 1$ jam)



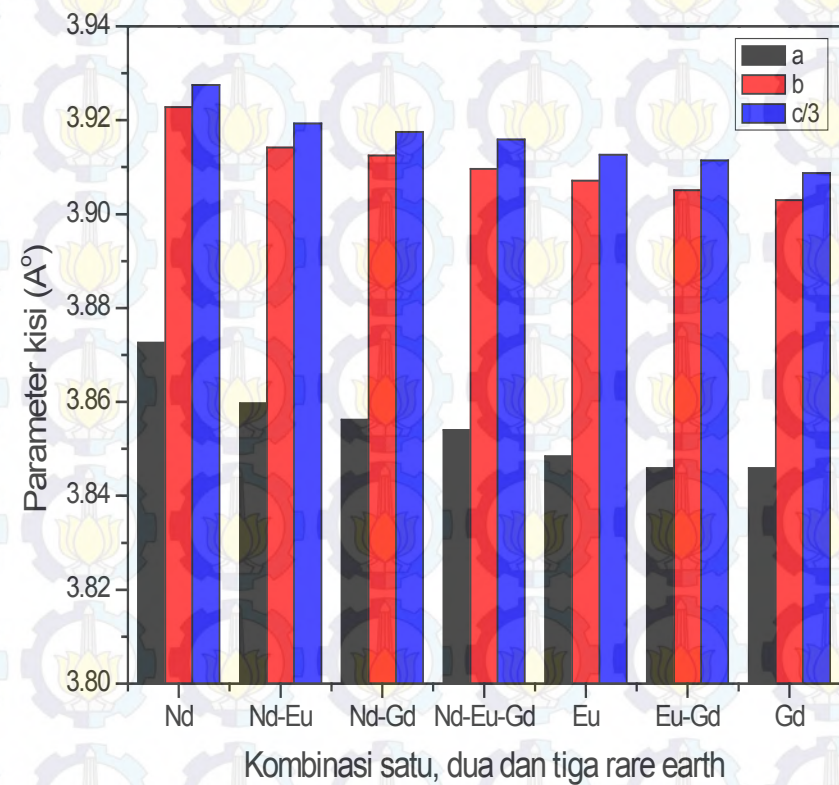
Nama	PDF No.	Senyawa	a (Å)	b (Å)	c/3 (Å)
Antipov, et.al	00-046-0229	NdBa ₂ Cu ₃ O _{6.88}	3,8799	3,9161	3,9215

Parameter Kisi NY-123 dan RE-123

Parameter kisi sampel NY-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

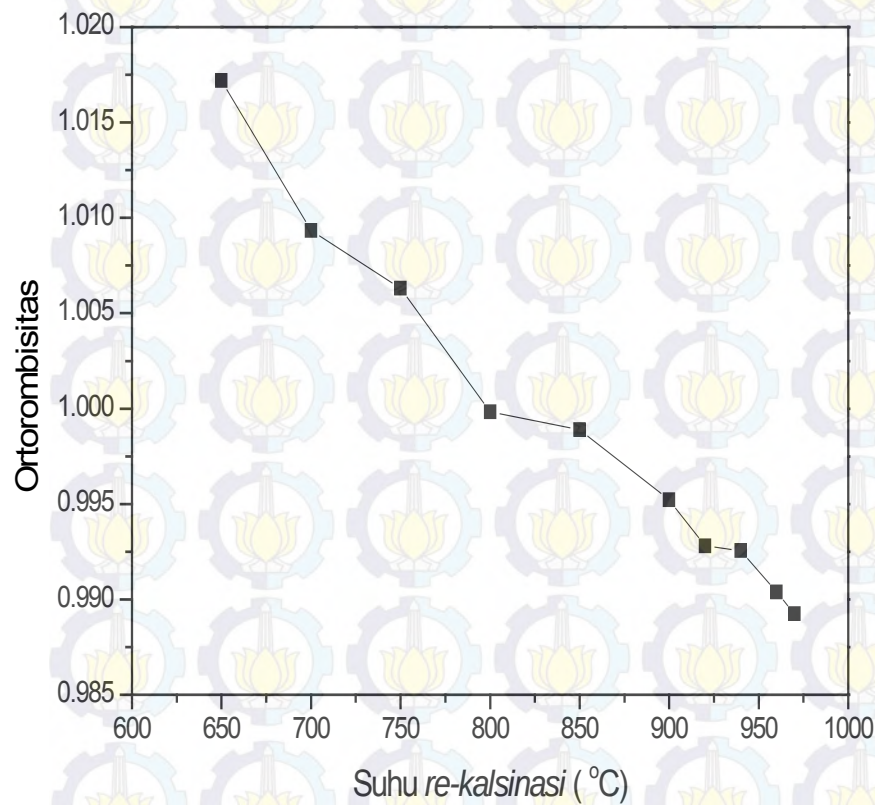


Parameter kisi sampel RE-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

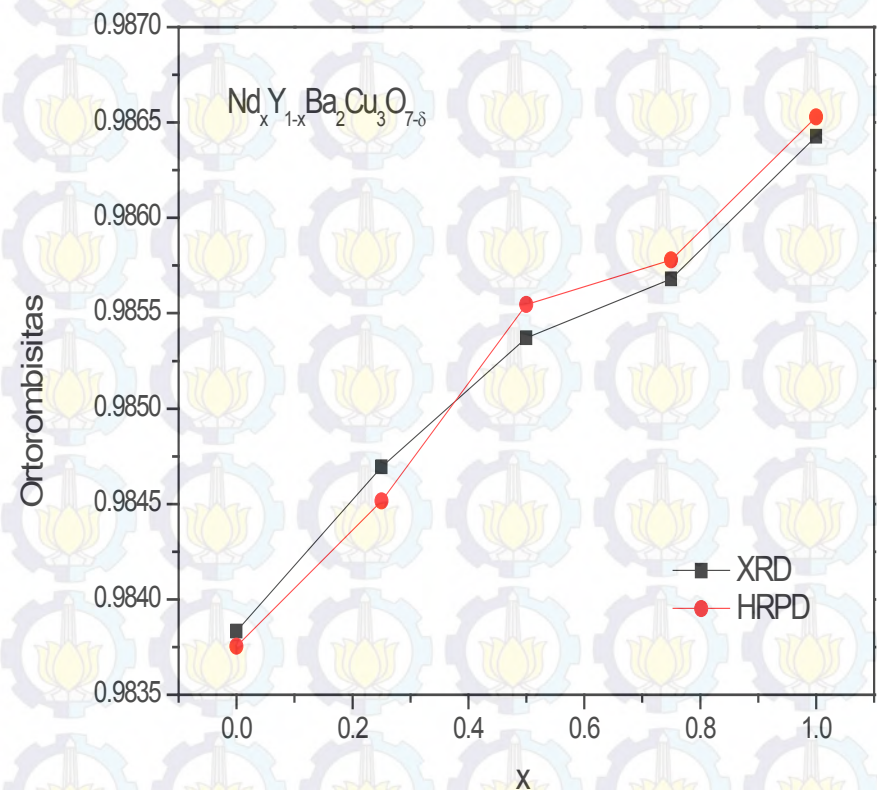


Ortorombisitas

Ortorombisitas sampel Nd-123
($T_{rc} = 650-970^{\circ}\text{C}$, $t_{rc} = 1$ jam)

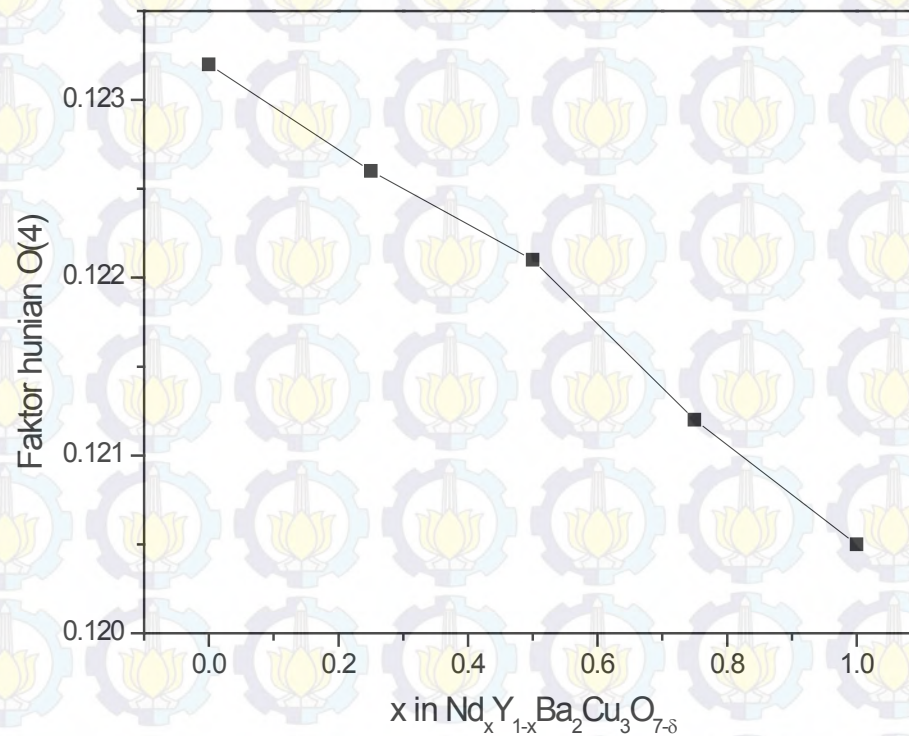


Ortorombisitas sampel $\text{Nd}_x\text{Y}_{(1-x)}-123$
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam) hasil
karakterisasi XRD dan HRPD



Faktor Hunian (Occupancy)

Faktor hunian (*Occupancy*) oksigen pada posisi O(4) dengan penambahan x dalam sampel $\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($T_{\text{rc}} = 970^\circ\text{C}$, $t_{\text{rc}} = 10$ jam)



Kandungan Oksigen

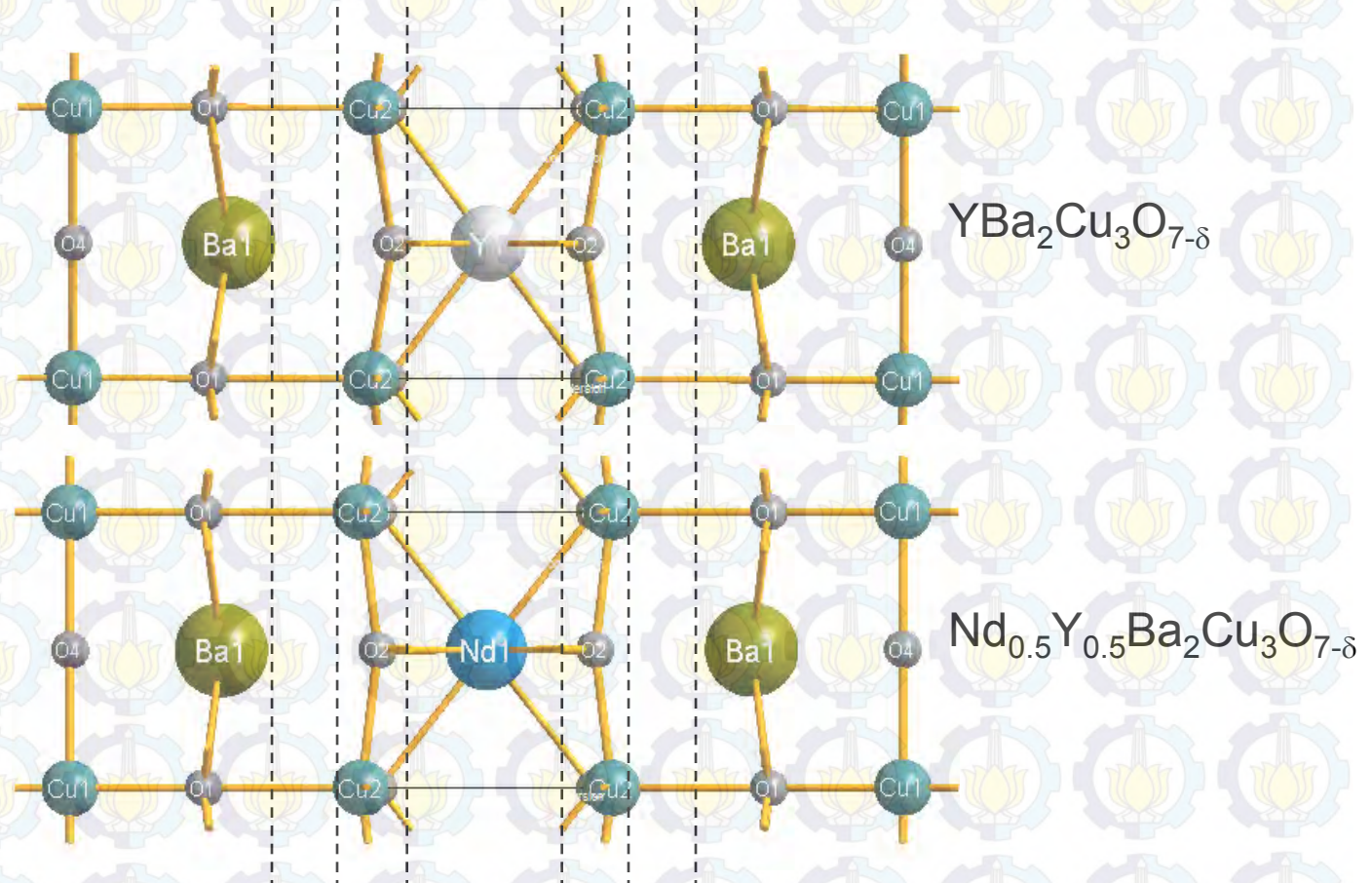
Kandungan oksigen sampel NY-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

Sampel	Kandungan Oksigen
$\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 0$)	$\text{YBa}_2\text{Cu}_3\text{O}_{6,97}$
$\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 0,25$)	$\text{Nd}_{0,25}\text{Y}_{0,75}\text{Ba}_2\text{Cu}_3\text{O}_{6,96}$
$\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 0,5$)	$\text{Nd}_{0,5}\text{Y}_{0,5}\text{Ba}_2\text{Cu}_3\text{O}_{6,95}$
$\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 0,75$)	$\text{Nd}_{0,75}\text{Y}_{0,25}\text{Ba}_2\text{Cu}_3\text{O}_{6,94}$
$\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ ($x = 1$)	$\text{NdBa}_2\text{Cu}_3\text{O}_{6,93}$

Occupancy (faktor hunian) oksigen pada posisi O1, O2, dan O3 dianggap penuh, sehingga jumlah oksigen pada posisi O1, O2 dan O3 adalah 6

Pergeseran Atom

Pergeseran atom-atom penyusun senyawa NY-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)



Jarak Antar Atom

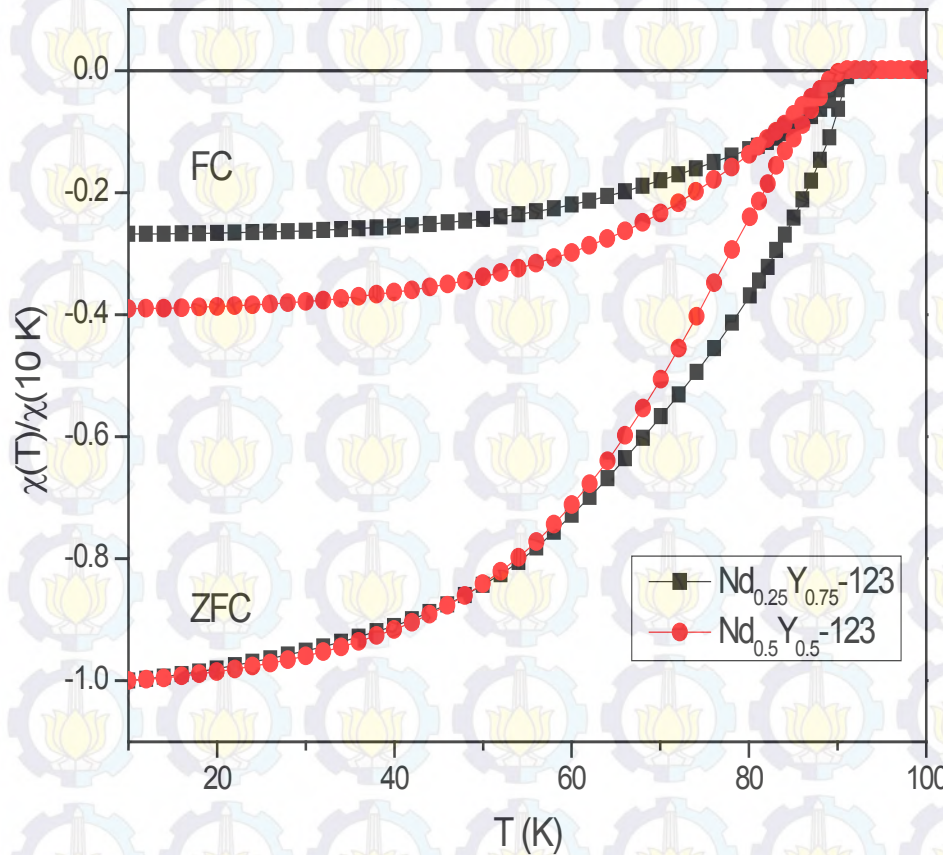
Jarak antar atom sampel NY-123
($T_{rc} = 970^{\circ}\text{C}$, $t_{rc} = 10$ jam)

Atom	Jarak antar atom ($\text{\AA}$) sampel $\text{Nd}_x\text{Y}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$				
	$x = 0$	$x = 0,25$	$x = 0,5$	$x = 0,75$	$x = 1$
Y/Nd-Ba	3,654	3,660	3,725	3,734	3,748
Y/Nd-O2	2,368	2,398	2,451	2,483	2,501
Y/Nd-O3	2,370	2,386	2,429	2,450	2,489
Y/Nd-Cu2	3,190	3,198	3,237	3,261	3,283
Ba-O1	2,739	2,748	2,749	2,756	2,764
Cu2-O2	1,935	1,939	1,941	1,943	1,947
Cu2-O3	1,959	1,967	1,970	1,975	1,983
Cu1-O4	1,911	1,916	1,923	1,934	1,947

SIFAT LISTRIK DAN MAGNETIK

Suhu Kritis (T_c) NY-123

Kurva M-T sampel NY-123
($T_{rc} = 970^\circ\text{C}$, $t_{rc} = 10$ jam)



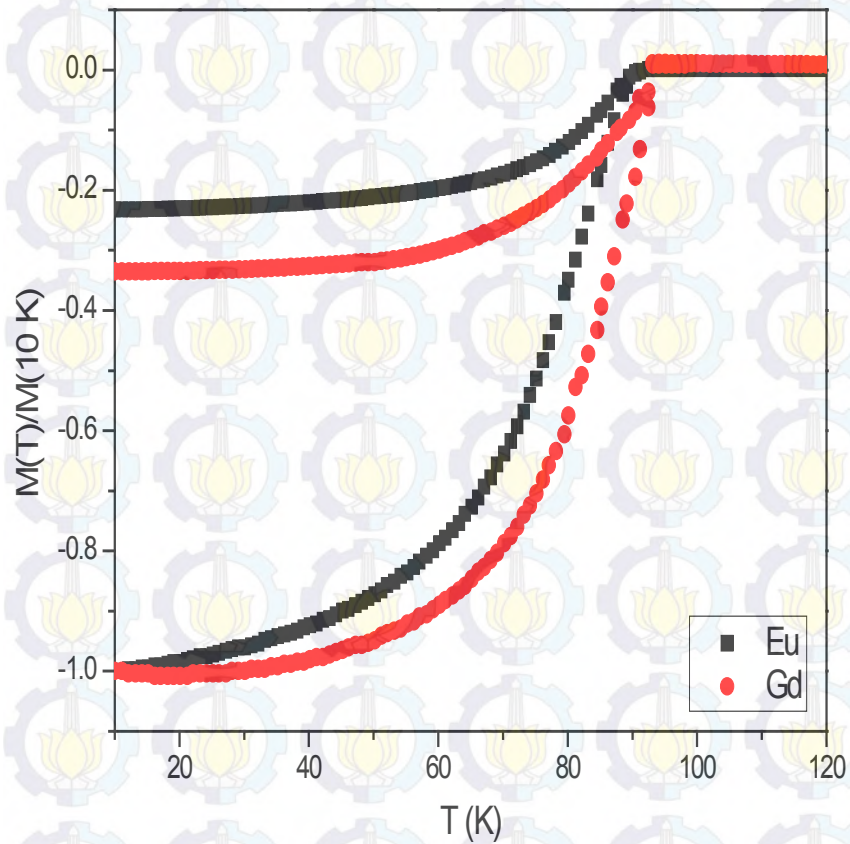
$T_c (\text{Nd}_{0.25}\text{Y}_{0.75}\text{-123}) \approx 91\text{ K}$

$T_c (\text{Nd}_{0.5}\text{Y}_{0.5}\text{-123}) \approx 92\text{ K}$

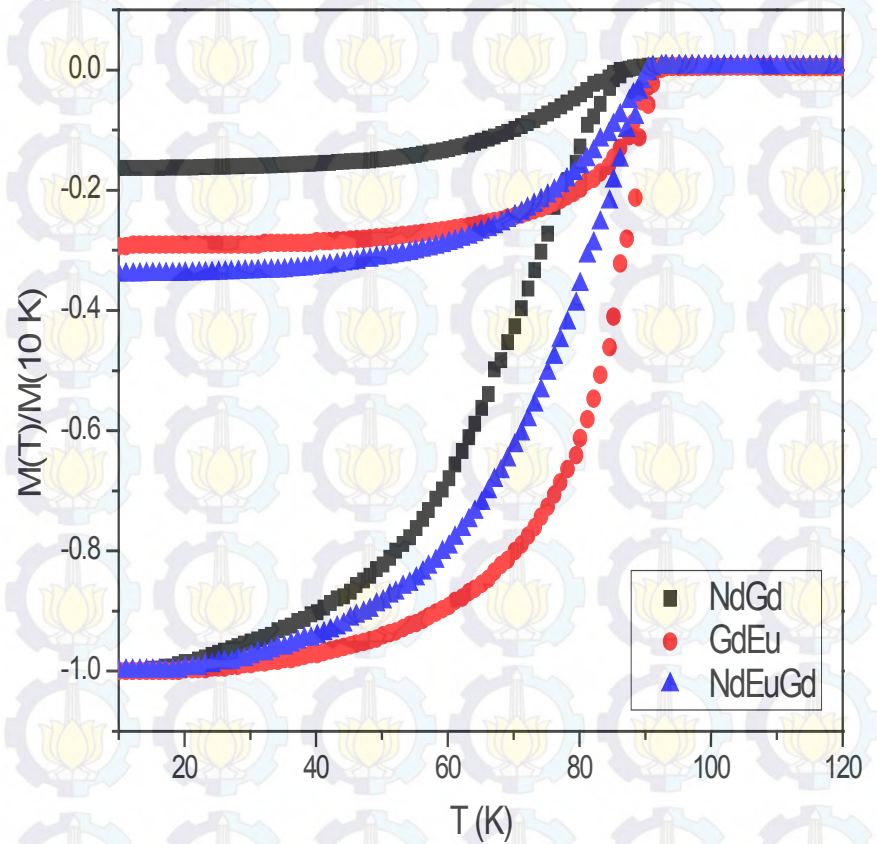
M-T SQUID dilakukan di
RIKEN (Issao Watanabe
dan Yasuyuki Ishii) dan
Chiba university (Hideto
Fukazawa dan Prof. Yoh
Kohori)

Suhu Kritis (T_c) RE-123

Kurva M-T sampel
Eu-123 dan Gd-123



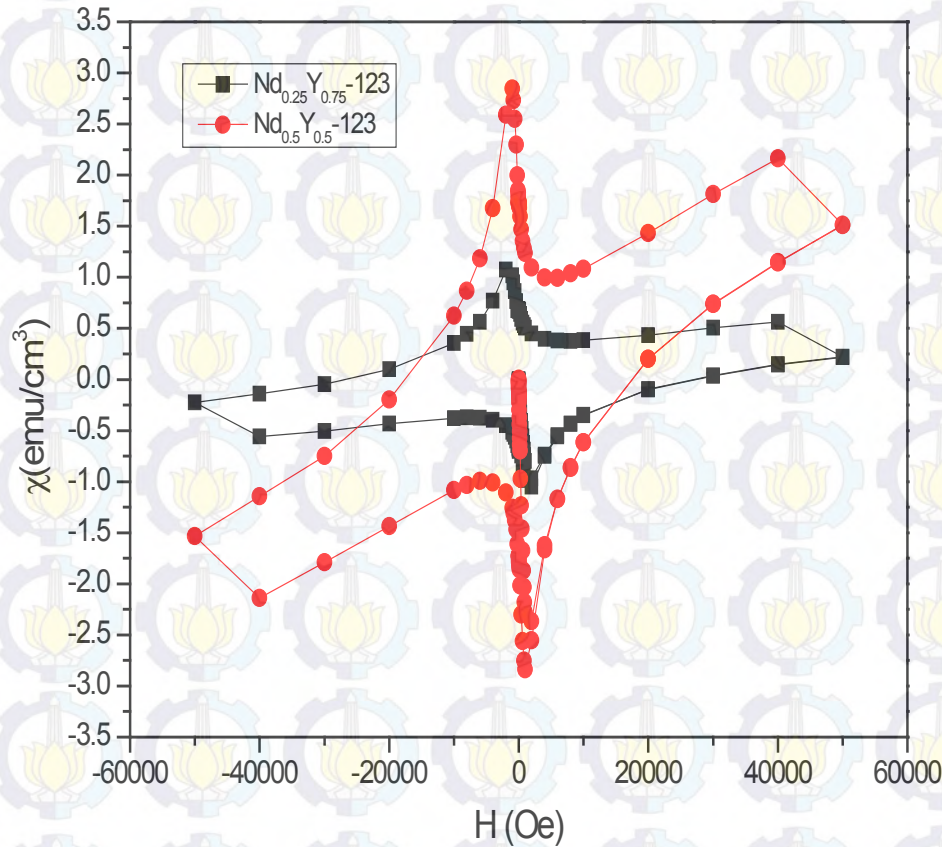
Kurva M-T sampel
NdGd-123, GdEu-123 dan
NdEuGd-123



$$88,5\text{ K} < T_c < 93,6\text{ K}$$

Rapat Arus Kritis (J_c)

Kurva M-H sampel NY-123
($T_{rc} = 970^\circ\text{C}$, $t_{rc} = 10$ jam)



Dengan menggunakan model Bean

$$J_c = \frac{3\Delta m}{2rV}$$

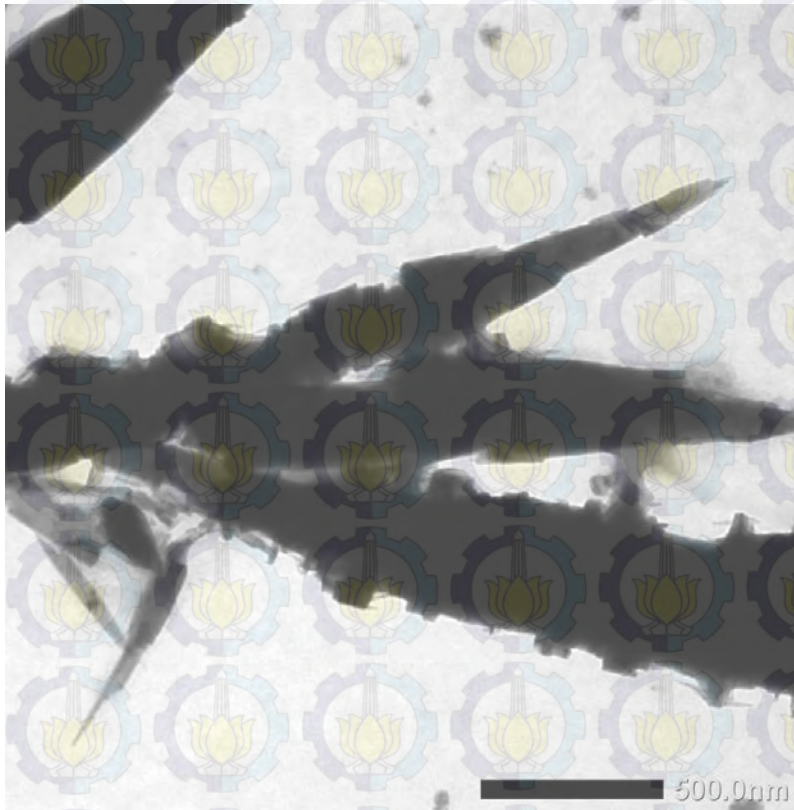
Harga rapat arus kritis sampel NY-123
($T_{rc} = 970^\circ\text{C}$, $t_{rc} = 10$ jam)

Sampel	J_c (A/cm ²)
$\text{Nd}_{0.25}\text{Y}_{0.75}\text{-123}$	4×10^4
$\text{Nd}_{0.5}\text{Y}_{0.5}\text{-123}$	1×10^5

M-H SQUID dilakukan di
RIKEN (Issao Watanabe
dan Yasuyuki Ishii)

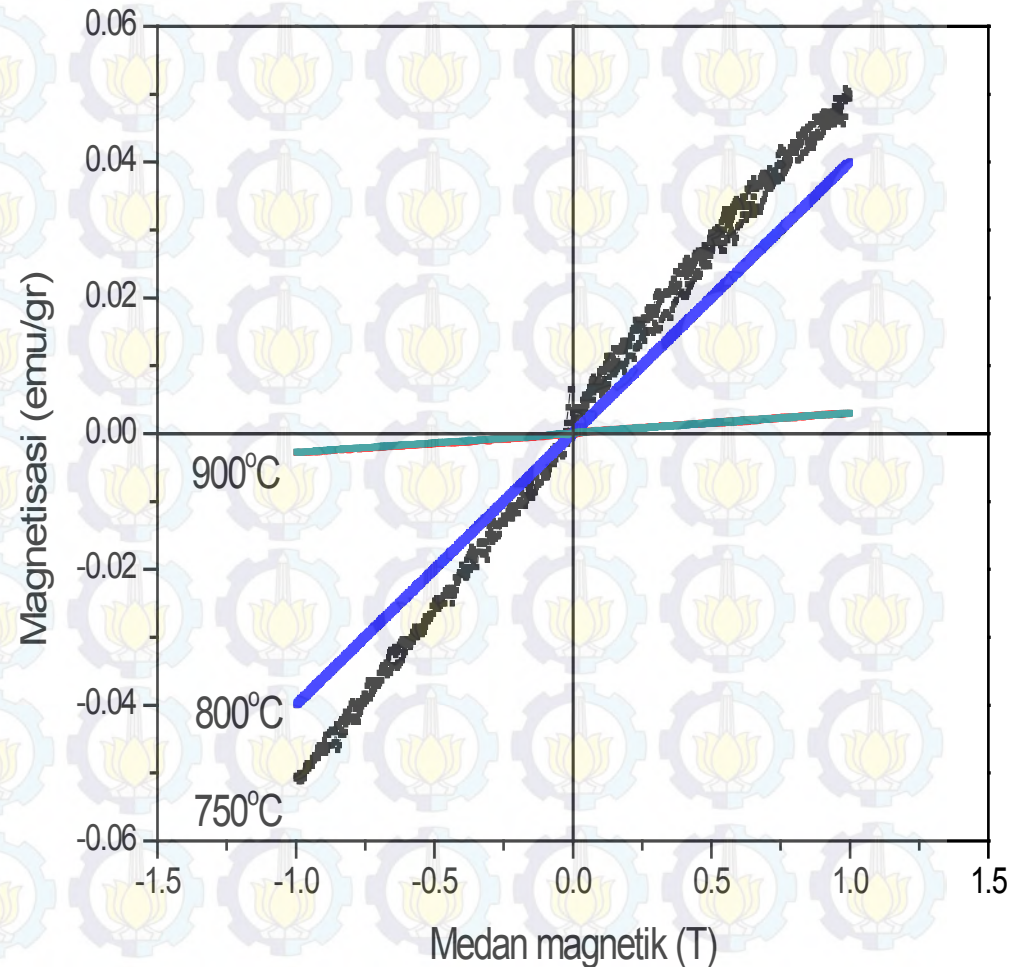
Paramagnetik

Gambar TEM sampel Nd-123
(Mixing 30 menit, $T_{rc} = 800^{\circ}\text{C}$,
 $t_{rc} = 1$ jam)



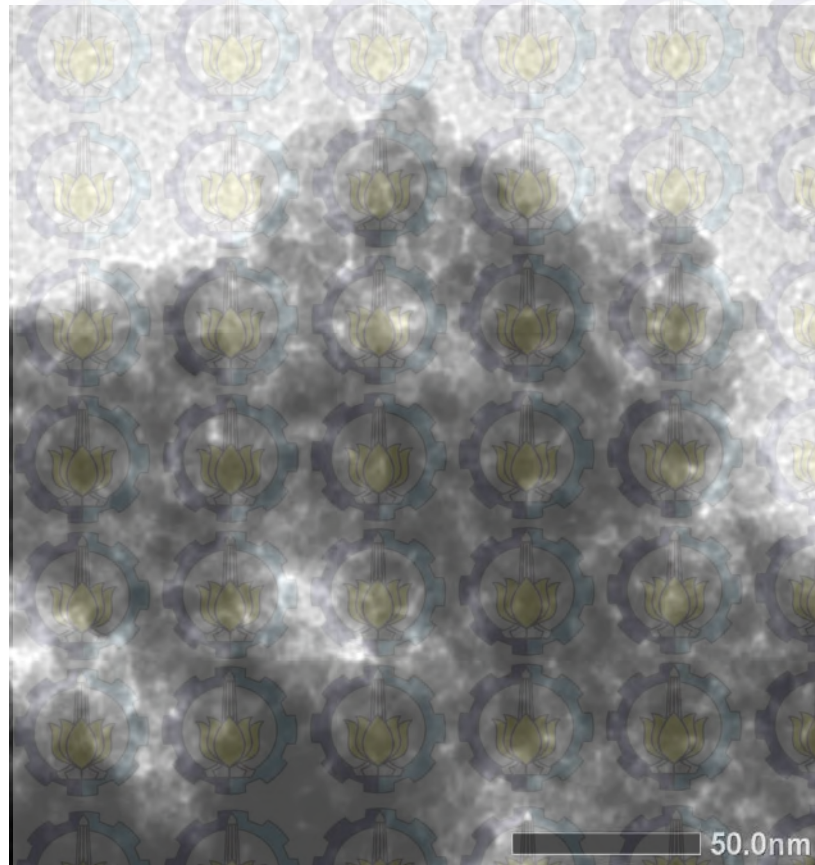
Ukuran partikel
> 100 nm

Karakterisasi VSM sampel Nd-123
(Mixing 30 menit, $T_{rc} = 750^{\circ}\text{C}$, 800°C ,
 900°C , $t_{rc} = 1$ jam)



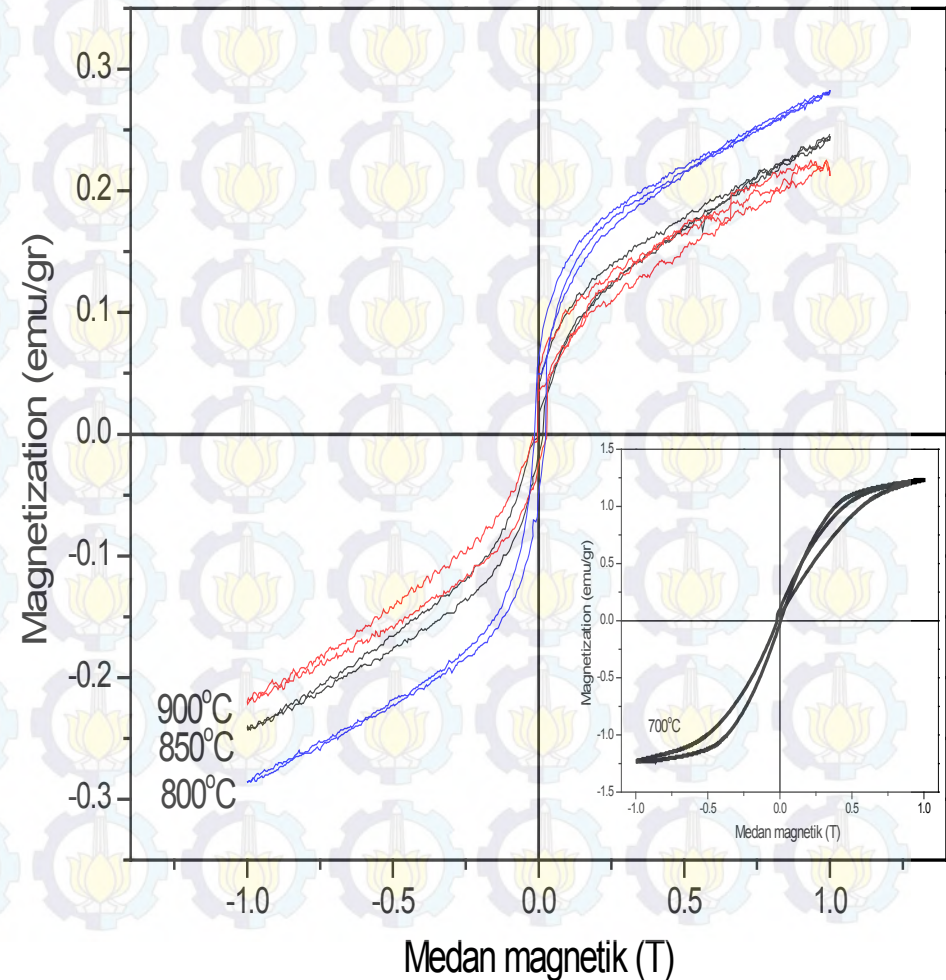
Ferromagnetik

Gambar TEM sampel NEG-123
(mixing 24 jam, $T_{rc} = 800^{\circ}\text{C}$,
 $t_{rc} = 30$ menit)

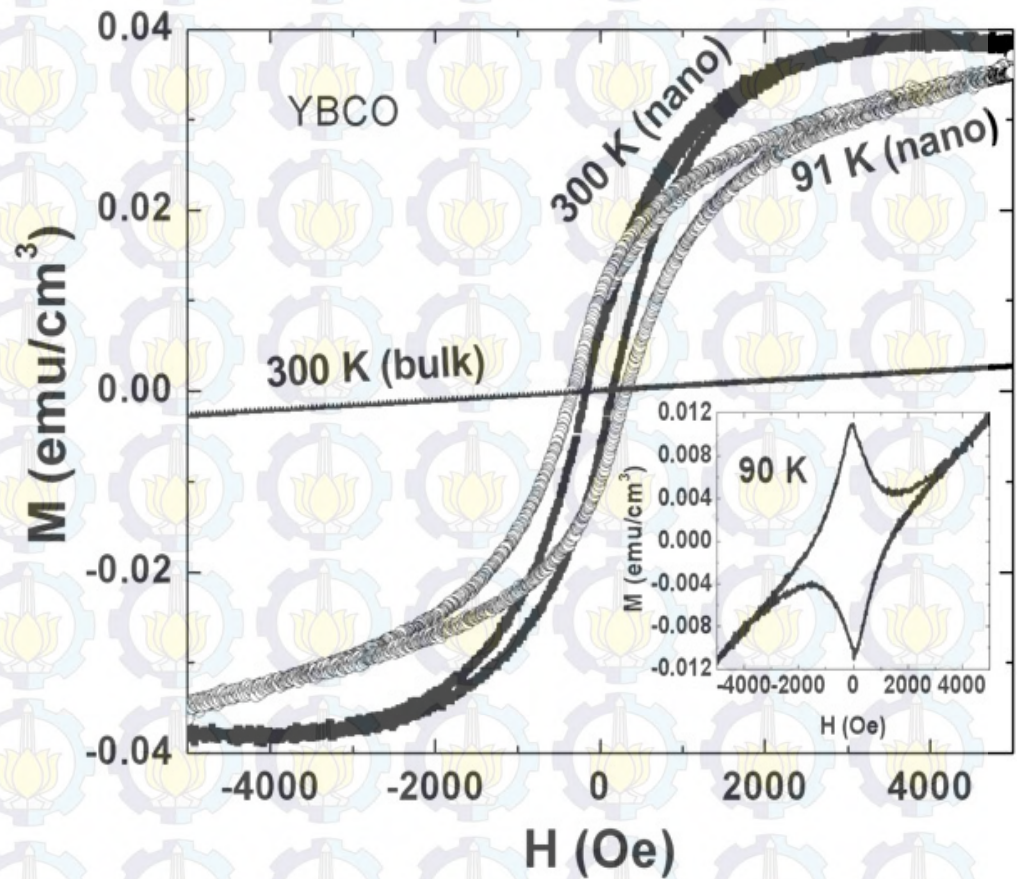
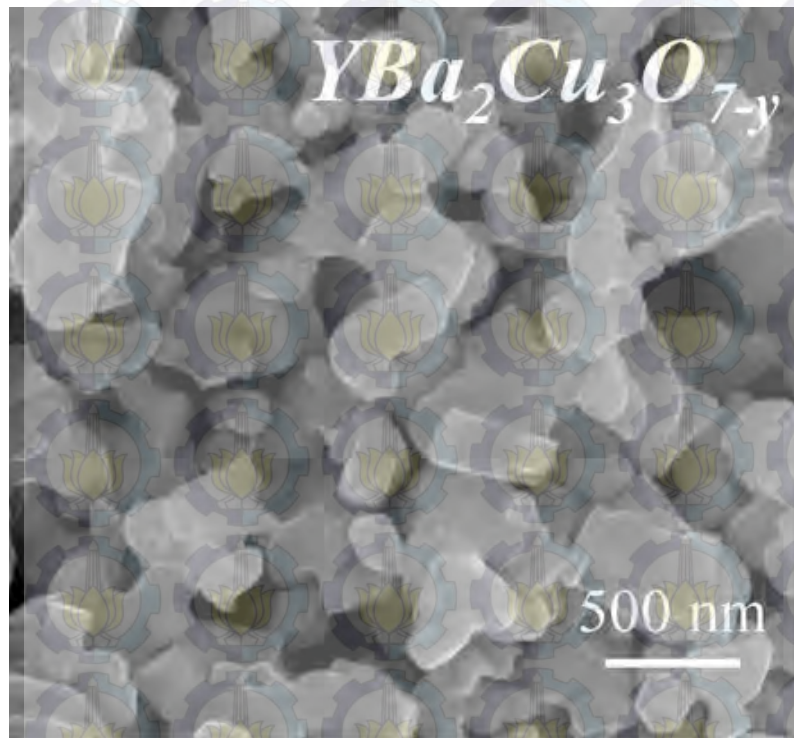


Ukuran partikel
< 50 nm

Hasil VSM sampel NEG-123
(Mixing = 24 jam, $T_{rc} = 800^{\circ}\text{C}$, 850°C ,
 900°C , $t_{rc} = 30$ menit)



Hasil penelitian Rao et.al.



KESIMPULAN

Kesimpulan

1. Proses sintesis superkonduktor $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ($\text{RE}=\text{Y}, \text{Nd}, \text{Eu}, \text{Gd}$) dengan *wet-mixing method* telah berhasil memperoleh kristal $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ dengan prosentase berat tertinggi sekitar 99,1 % (mendekati fasa tunggal).
2. Penambahan unsur Nd pada superkonduktor $\text{Nd}_x\text{Y}_{(1-x)}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ dengan $x = 0, 0,25, 0,5, 0,75$ dan 1 menunjukkan bertambahnya nilai parameter kisi, volume sel satuan dan ortonombisitas. Urutan nilai parameter kisi, volume sel satuan dan ortonombisitas dari yang terkecil sampai terbesar adalah Y-123, $\text{Nd}_{0,25}\text{Y}_{0,75}$ -123, $\text{Nd}_{0,5}\text{Y}_{0,5}$ -123, $\text{Nd}_{0,75}\text{Y}_{0,25}$ -123 dan Nd-123. Penambahan unsur Nd juga mengakibatkan migrasi oksigen yang diperlihatkan dengan berkurangnya faktor hunian oksigen (*occupancy*) pada posisi O(4). Penambahan unsur Nd pada superkonduktor $\text{Nd}_x\text{Y}_{(1-x)}$ -123 dapat meningkatkan nilai suhu kritis, rapat arus kritis dan sifat ketahanan superkonduktor terhadap medan magnet. Suhu kritis dan rapat arus kritis sampel $\text{Nd}_{0,25}\text{Y}_{0,75}$ -123 masing-masing adalah 90,5 K dan $4 \times 10^4 \text{ A/cm}^2$, sedangkan suhu kritis dan rapat arus kritis sampel $\text{Nd}_{0,5}\text{Y}_{0,5}$ -123 masing-masing adalah 91,5 K dan $1 \times 10^5 \text{ A/cm}^2$.
3. Kombinasi satu, dua dan tiga unsur RE (Nd, Eu, Gd) pada superkonduktor $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ menghasilkan perubahan nilai parameter kisi serta volume sel satuan, dengan urutan dari terbesar sampai terkecil, yaitu Nd-123, $\text{Nd}_{0,5}\text{Eu}_{0,5}$ -123, $\text{Nd}_{0,5}\text{Gd}_{0,5}$ -123, $\text{Nd}_{0,33}\text{Eu}_{0,33}\text{Gd}_{0,33}$ -123, Eu-123, $\text{Eu}_{0,5}\text{Gd}_{0,5}$ -123 dan Gd-123. Urutan tersebut merupakan kebalikan dari pergeseran sudut 2θ yang diakibatkan oleh perubahan nilai parameter kisi. Suhu kritis dan sifat ketahanan superkonduktor terhadap medan magnet superkonduktor Gd-123 (93,6 K) lebih besar dibandingkan superkonduktor Eu-123 (92,5 K). Penambahan Nd pada kombinasi dua dan tiga RE dapat menurunkan suhu kritis dan sifat ketahanan superkonduktor pada medan magnet dengan urutan dari terbesar sampai terkecil adalah $\text{Eu}_{0,5}\text{Gd}_{0,5}$ -123 (93,1 K), $\text{Nd}_{0,33}\text{Eu}_{0,33}\text{Gd}_{0,33}$ -123 (92 K) dan $\text{Nd}_{0,5}\text{Gd}_{0,5}$ -123 (88,5 K).
4. Proses sintesis dengan *wet-mixing method*, serta perlakuan *mixing* selama 24 jam telah menghasilkan nano partikel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$. Sampel $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ dengan ukuran partikel lebih kecil dari 50 nm memperlihatkan sifat feromagnetik pada suhu ruang, sedangkan sampel dengan ukuran partikel lebih besar dari 100 nm memperlihatkan sifat paramagnetik.



TERIMA KASIH