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RESEARCH ARTICLE

STRUCTURAL, ELECTRICAL AND THERMAL ANALYSIS OF $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ SUPERCONDUCTORS.

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Abstract

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(Y,Eu)-Ba-Cu-O samples were prepared by solid state method of heat treatment for obtaining single phase materials. The relationship between Y-Eu substitution and the superconducting properties of $Y_{1-x}Eu_xBa_2CuO_{7-\delta}$ ($0.0\leq x \leq 0.9$) solid solution have been studied.
DC resistance measurements showed that Eu substitution was affected the normalized resistance and T_c values. X-ray diffraction analysis showed that the main phase in the prepared (Y-Eu)–Ba-Cu-O samples was the orthorhombic phase. The samples were tended to form plate type of single crystals with preferential orientation in c-direction.
The crystallite size of the prepared samples was calculated from X-ray diffraction patterns using Scherer's equation and has a value of 21nm for pure YBCO and that of Eu content up to 0.9 samples.
Thermal analysis of (Y,Eu)-Ba-Cu-O system were performed by means of TG and DTA. The oxygen content was estimated from TG-analysis and it was found to decrease with increasing Eu-content. DTA showed three-regions which were classified as follows:
(1) Exothermic peak as a result of chemically bonded dehydration temperature.
(2) Endothermic peak due to tetragonal orthorhombic transformation temperature.
(3) Exothermic peak which was related to melting temperature.

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Introduction:-

Since the discovery of YBCO, a high temperature superconductor (HTS) has gathered a lot of attention because of its potential in many applications such as levitated systems, magnetic shields, electronics and communications [1]. It is known that the properties of YBCO superconductors are improving by replacing Y with other light rare earth elements [2]. Recently, considerable attention has been devoted to the superconducting properties of LRE123 systems [3-6], (where LRE: light rare earth elements like Nd,Sm,Eu and Gd). Generally unlike Y123 which forms only a stoichiometric compound, light-rate-earth elements form a solid solution in LRE123 systems and this solid solution leads to a variation in superconducting properties.

(LRE)Ba-Cu-O superconductors have revealed promising performance, such as high T_c, J_c and irreversible magnetic fields than in Y-Ba-Cu-O [7-8].

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$Y_{1-x}Sm_xBa_2Cu_3O_{7-\delta}$ and $Y_{1-x}Nd_xBa_2Cu_3O_{7-\delta}$ solid solution superconductors have been reported [9]. In the present work a detailed study has been performed for the $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ system. The crystal growth and the superconductivity of the prepared samples are studied by DC resistance, thermal analysis (TG-DTA) and X-ray diffraction (XRD) techniques.

Experimental Work:-

Samples Preparation

Samples with nominal composition of $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ (x = 0.0, 0.1, 0.3, 0.5, 0.7 and 0.9) were prepared through a solid state heat treatment method using high purity powders (≥ 99.99%) of Y_2O_3, Eu_2O_3, $BaCO_3$ and CuO. The mixture of starting materials was calcined in alumina crucibles, firstly at 900°C for 24hr, secondly at 920°C for 60hr, then grounded, pressed into pellets and finally treated by a heat cycle as follows:

- Heated from room temperature to 920°C and kept at this temperature for 12hr in air.
- Cooled from 920°C to 680°C in 4hr with following O_2.
- Kept at 680°C for 10hr with O_2 flowing.
- Cooled from 680°C to 400°C in 4h in air.
- Kept at 400°C for 10hr in air.
- Cooled from 400°C to room temperature in the furnace.

The preliminary test of Meissner effect showed that all samples are superconductors at liquid nitrogen temperature (77K).

Samples Characterization:-

The characterization of the prepared samples was carried out by:

1- Electrical DC resistance measurements for T_c determination by the standard 4-probe method with silver contacts.

2- X-ray diffraction (XRD) for phase identification by X'pert type Philips Diffractometer2000 with a wavelength of (Cu - Kα , λ = 1.54056 A°).

3- Thermo-Gravimetric technique (TG): the temperature variation of weight loss was measured by TGA–50H, detector by using about 11-15mg of samples of YEu(x)Ba-Cu-O in a Pt-cell and the atmosphere was air. The cooling rate was 20°C/min in the temperature range from 800°C to 25°C.

4- Differential thermal analysis (DTA): A Shimadzu DTA–50 was used for DTA measurements with a cooling rate of 20°C/ min for all samples with mass 0.01mg, the atmosphere is nitrogen with rate flow 20 ml/min in the temperature range from 1200°C to 25°C.

Results and Discussion:-

The normalized resistance to the value at 300K were recorded for $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples. All samples have superconducting behavior as shown in Fig (1). The transition temperature T_c at zero resistance was evaluated for the prepared samples and was varied from 92K to 84K depending on Eu content.

Eu ions are in a trivalent state Eu^{3+} at regular occupying Y^{3+} sites and also can substituted at Ba^{2+} sites [10]. The variation in T_c was attributed to the double substitution of Eu^{3+} at both Y^{3+} and Ba^{2+} sites with different probabilities.

X-ray diffraction (XRD) patterns of the prepared samples were illustrated in Figs (2,3). The pure YBCO has orthorhombic 1-2-3 structure which is responsible for superconducting phase. The characteristic peaks of this phase were indexed and it was obtained that the strongest peaks are (013) and (103). The relative intensities of (001) and (hkl) planes were affected due to Eu substitution in YBCO as shown in Figs (2,3) and table (1). This was due to the preferential orientation in c–direction. The (003), (005) and (006) planes were grown due to the fast growth rate in a, b directions and slow growth rate along c-direction. In the Y(Eu)BCO samples, the resulting X-ray patterns were affected for instance, the lines (003), (005) and (006) were amplified while the lines (013) and (103) were depressed. The samples of Y(Eu)BCO system were tended to form plate type of single crystals with orthorhombic phase. X-ray diffraction analysis was confirmed that the main phase in $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples was the orthorhombic structure.

The unit cell parameters were affected as indicated in table (2) due to Eu substitution in Y–sites and explained as follows:

1-For Eu contents in the range ($0.1 \le x \ge 0.5$), the unit cell volume was decreased**.**

Note: ionic radius of Ba^{2+} equals 1.42A°, for Y^{+3} equals 1.019A°, and that of Eu^{+3} equals 1.066A° [10]. The decrease in the unit cell volume was due to Eu^{3+} substitution at Ba^{2+}sites in addition to original Eu^{3+} substitution at Y^{3+} sites.

2-In the range ($0.5 \le x \le 0.9$) for the $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples, the lattice parameters a, b and c were increased and so the unit cell volume was increased for these samples. This result was due to Eu^{3+} substitution at Y^{3+}sites were more probable and Eu^{3+} substitution at Ba^{2+} sites was minimized in this range of Eu contents.

The crystallite size (t) of the prepared samples was calculated from X-ray analysis using Scherer's equation [11]:-
$t = (0.9\lambda / \beta \cos \theta)$

Where λ is the X-ray wavelength, β is the peak width at half maximum and θ corresponds to the peak position. The calculated crystallite size (t) value of 21nm for pure YBCO and samples of Eu content up to 0.9.

Fig (4) showed TGA curves recorded in the temperature range from 25°C to 800°C in air for $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples with x values ranging from x =0.0 to x = 0.9.

The initial weight was taken as a standard value. The variation of initial weight value was plotted as a function of temperature. It was noted that the oxygen content of the $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples were decreased with increasing Eu content (x). The oxygen content was decreased from 6.95 for x =0. 0 to 6.83 for x = 0.9.

The differential thermal analysis DTA for the prepared samples were recorded with a cooling rate 20°C/ min. Figs (5,6) showed DTA for $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples which can be classified into three-regions:
1-First region: Exothermal process at temperature T_1 ranging from 200°C to 280°C as a result of loss of chemically bonded water dehydration and dissociation of hydroxides.
2-Second region: Endothermic process at temperature T_{t-0} and was varied from 570°C to 800°C. This is due to tetragonal orthorhombic transformation for $Y_{1-x}Eu_x123$ superconductors.
3-Third region: Exothermal process at T_m related to the melting temperature and have values ranging from 975°C to 1025°C. The results of DTA were summarized in table (3).

Table (1):- Relative intensities of important peaks in the orthorhombic system of $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$

Plane (hkl)	**I_r for $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$**					
	x = 0.0	**x = 0.1**	**x = 0.3**	**x = 0.5**	**x = 0.7**	**x = 0.9**
003	16.99	49.06	37.59	36.79	42.59	54.25
013	50.53	32.94	25.83	19.71	32.59	55.56
103	100	43.26	33.11	29.59	56.37	88.03
005	22.27	71.58	60.39	51.68	47.31	43.33
006	31.16	100	100	100	100	100

Table(2):- The unit cell parameters and its volume of $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples from X-ray diffraction analysis.

Eu content (x)	**Unite cell parameters**			**Volume $(A°)^3$**
	a (A°)	**b (A)°**	**c (A°)**	
0.0	3.821	3.890	11.689	173.741
0.1	3.820	3.880	11.689	173.250
0.3	3.818	3.877	11.689	173.025
0.5	3.823	3.883	11.694	173.594
0.7	3.832	3.899	11.712	174.988
0.9	3.835	3.894	11.712	174.901

Table(3): Summarizing of the DTA results for the three regions and the corresponding temperatures T_1, T_{t-o} and T_m as a function of Eu content (x) in $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$.

Eu Content (x)	**T_1(°C)**	**$T_{t\to o}$ (°C)**	**T_m(°C)**
0.0	250	600	975
0.3	260	780	1025
0.5	280	800	1020
0.9	200	570	1010

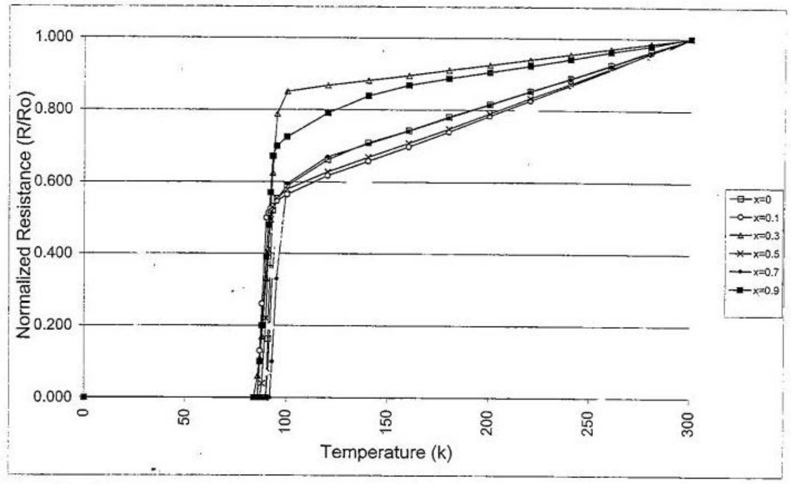


Fig 1

Fig (1) : The normalized resistance (R/R_o) as a function of temperature T(K) of $Y_{1-x}Eu_x\ Ba_2Cu_3O_{7-\delta}$ superconductors for x=0.0,0.1,0.3,0.5,0.7, and 0.9

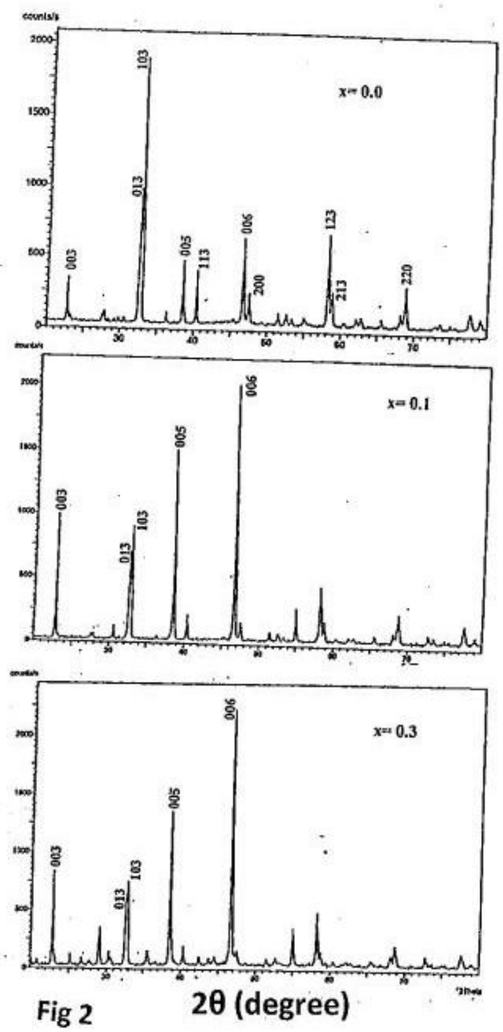


Fig 2

Fig (2) : X-ray diffraction XRD patterns for $Y_{1-x}Eu_x$ $Ba_2Cu_3O_{7-\delta}$ samples with x=0.0,0.1,and 0.3

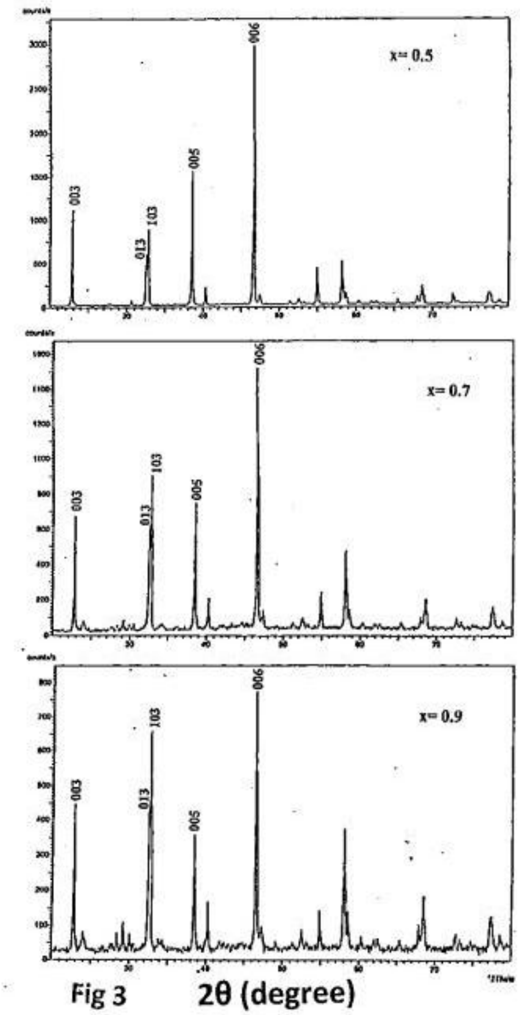


Fig (3) : X-ray diffraction XRD patterns for $Y_{1-x}Eu_x$ $Ba_2Cu_3O_{7-\delta}$ samples with x=0.5,0.7,and 0.9

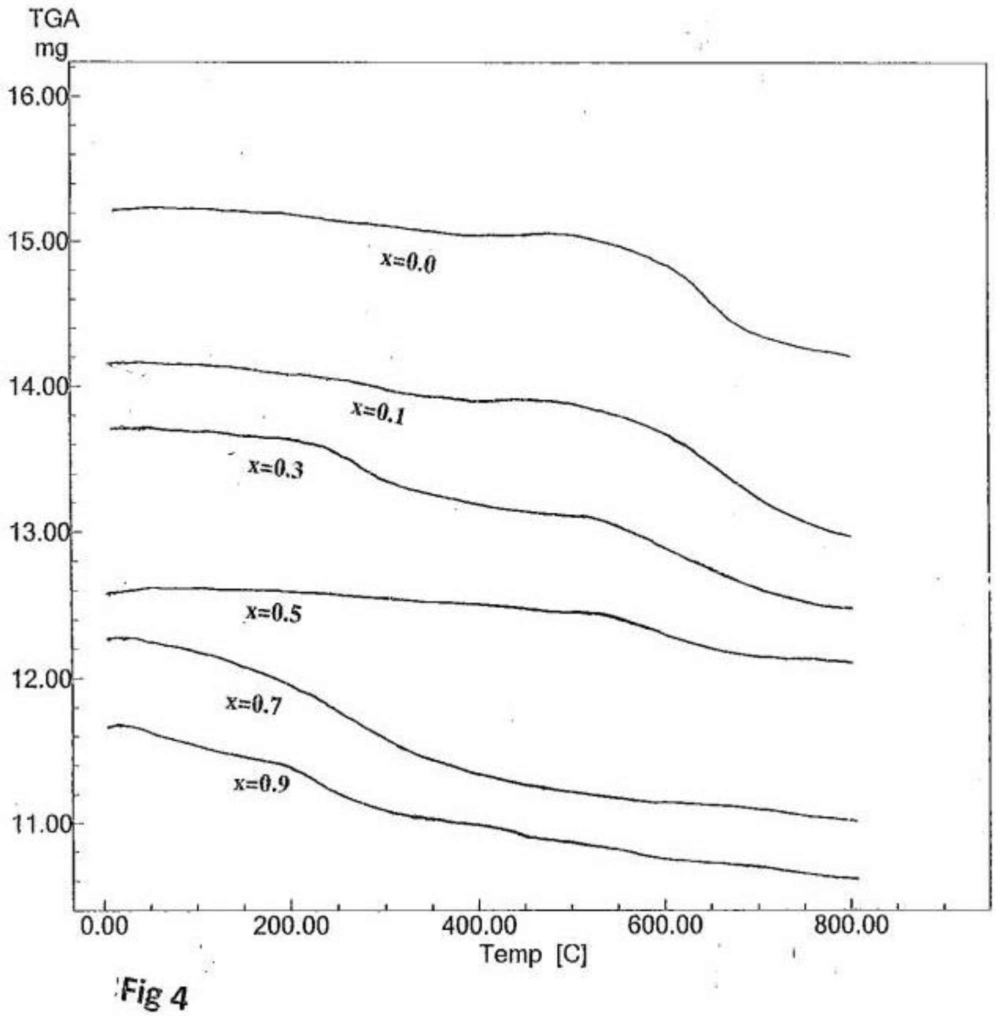


Fig (4) : Thermo-gravimetric analysis TGA, the mass percentage change as a function of temperature for $Y_{1-x}Eu_x Ba_2Cu_3O_{7-\delta}$ samples.

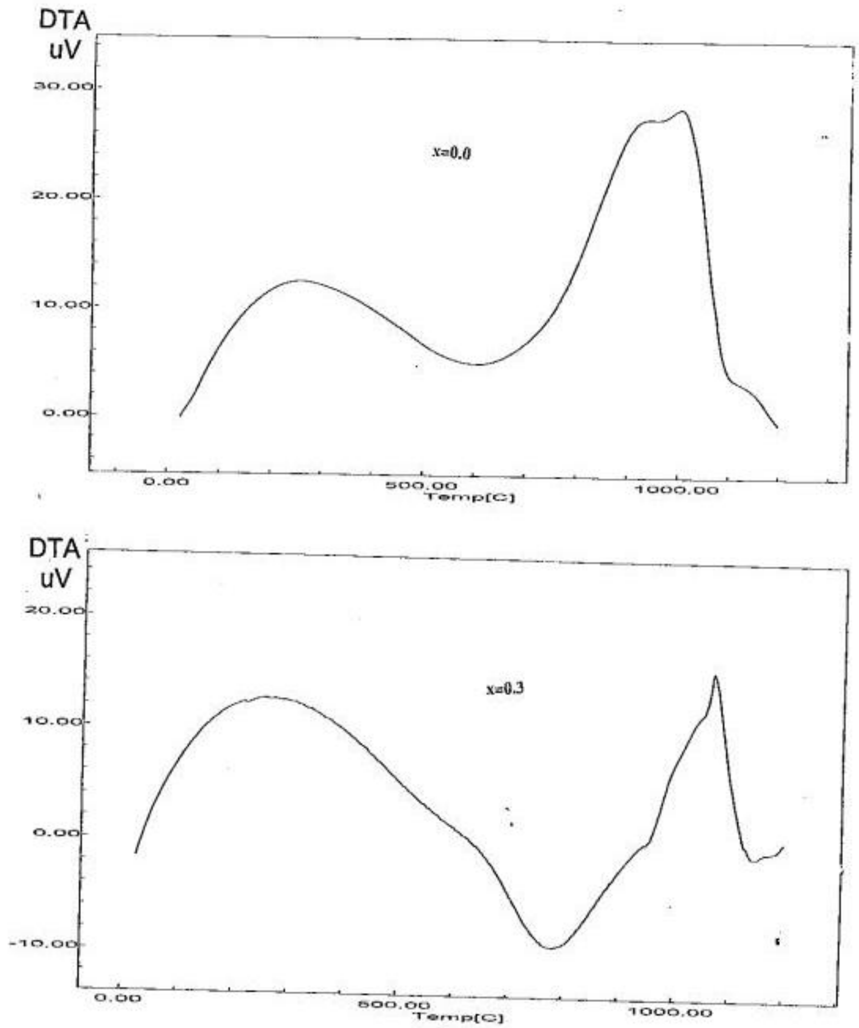
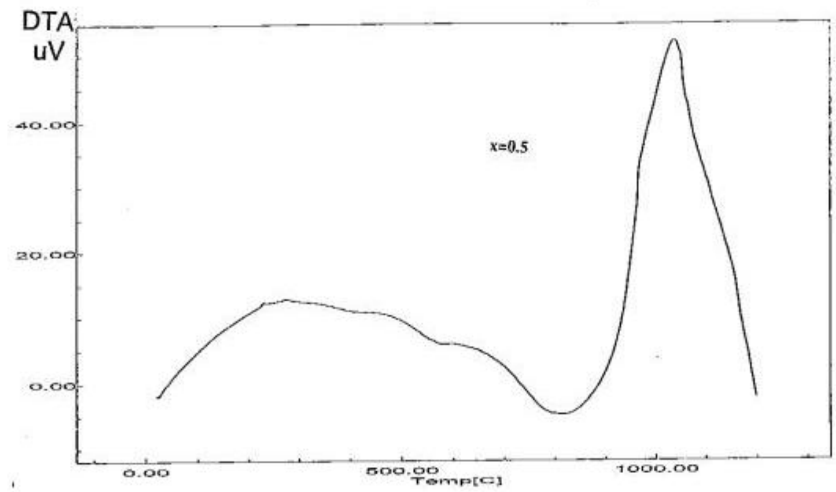


Fig 5

Fig (5) : Differential thermal analysis of $Y_{1-x}Eu_x Ba_2Cu_3O_{7-\delta}$ for samples (x=0.0 and x=0.3).



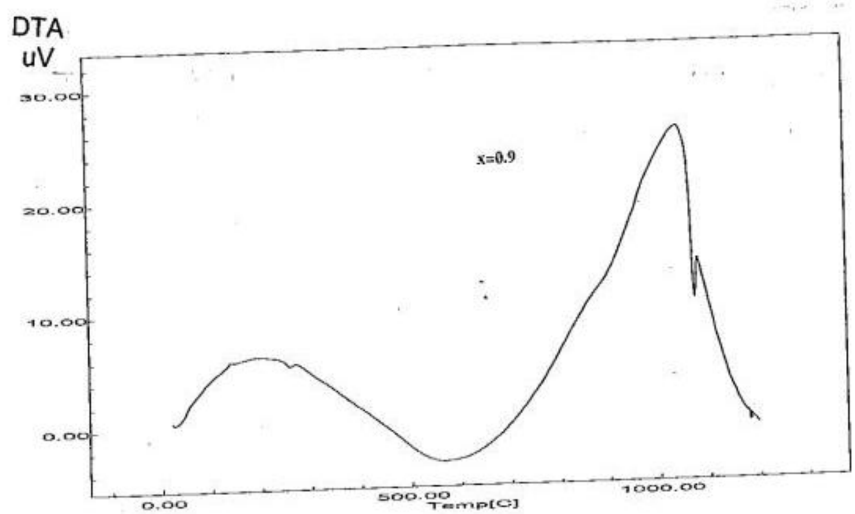


Fig 6

Fig (6) : Differential thermal analysis of $Y_{1-x}Eu_x Ba_2Cu_3O_{7-\delta}$ for samples (x=0.5 and x=0.9).

Conclusion:-

1- $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ samples tending to form plate type of single phase with preferential orientation in c-direction and the crystallite size of these samples equal to 21nm.

2- The oxygen content was estimated from TG analysis and it was found to decrease with increasing Eu content.

3- Unlike Y123 which forms only one a stoichiometric compound, the light rare earth element like Eu substitution in Y sites form a solid solution of $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ and each sample has a unique superconducting transition and also has a unique TG and DTA thermal behavior.

4- It was found that the melting temperature (T_m) of the $Y_{1-x}Eu_xBa_2Cu_3O_{7-\delta}$ composites was increased in samples contained the two different rare earth elements Y and Eu.

5- The mixture of the two different rare elements Y-Eu was very uniform and all samples showed a superconducting phase with a sharp transition and orthorhombic structure which is necessary for the presence of superconducting phase.

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