

Precursor Size Effects on Kinetics Parameters of Bi-2223 Growth

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Abstract: The Bi-2223 phase growth was studied on the view growth Kinetics and described by using suggested kinetic equations, and the kinetics parameters of the 2223 growth were experimentally determined. The precursor particle size effect on Bi-2223 phase growth was studied, as well. Precursors in this experiment were made by four different techniques with different particle sizes, and samples were sintered at same temperature for different sintering time. For the four different precursors, the amount of 2223 phase in the reactant was determined by analysis of X-ray diffraction. Experimental results were analyzed in the view of 2223 phase growth kinetics. Particle size dependence of the mean lifetime of 2212 and 2223 phases were studied. And the effects of special area of precursor particle on 2223 crystal growth were also analyzed. The velocity of the 2223 phase growth was investigated, which was found to be dependent on both the precursor particles' diameter and its total surface area. The velocity of the 2223 phase decomposition was determined, as well. The optimum sintering time t_{\max} and maximum amount of 2223 phase were calculated, and they were found to be dependent on the mean lifetime of 2212 and 2223 phases. When the particle size decreases, the optimum sintering time decreases, and the maximum amount of 2223 phase increases.

Keywords: Bi-2223 phase, growth, precursor, particle size, kinetics.

1. INTRODUCTION

Among high temperature superconducting cuprates, the 2223 phase of Bi-based system has a critical temperature as high as 110 K. The 2223 phase with a stoichiometry of $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$, synthesized at high temperature is a complex compound. The growth morphology of the 2223 phase with solid state reactions mainly depends on synthesis methods, in addition to the properties of precursors and sintering processing [1-15]. Phase development and kinetics of the 2223 phase relate to the synthesis processing [16-25], as well. Properties of precursor particles, especially grain size of particles, are important factors to obtain high quality 2223 crystals. The purpose of this paper is to quantitatively describe the 2223 phase growth by using suggested kinetic equations, and furthermore to determine the kinetics parameters of the 2223 growth. As known, the rate of 2223 phase growth is dependent on its preparation processing. Researchers can prepare fine 2223 phase samples with some special processing, in which the techniques of an intermediate grinding and re-sintering was popular and often to be sued. However, on the view of growth kinetics, an intermediate grinding will break the grown crystals and re-sintering can cause the ground crystals continue to growth at new conditions. For the kinetics study, the above popular

techniques are not a pure processing, which can not show the original growth of the 2223 phase. To study the growth kinetics, an original processing of the 2223 phase growth was performed, in which the 2223 phase growth, from starting materials to its appearance and changes, was recorded. In the original experiment no decorated or extra processing was added.

In our experiment, the starting materials were different kinds of precursors with different particle sizes. And the effect of particle size of precursors on the Bi-2223 phase growth was studied, as well. In this experiment, precursors were made by four different techniques with different particle sizes and sintered at the same temperature. Precursor particle sizes were determined by a low-angle laser light scattering device. Amount of 2223 phase for the different precursor was determined by analysis of X-ray diffraction (XRD). Experimental results were analyzed in the view of 2223 phase growth kinetics. For the four precursors, particle diameters dependence of the mean lifetime of 2212 and 2223 phases were determined, and denoted as τ and τ_1 . And the effects of special area of precursor particle on 2223 crystal growth were analyzed, as well. The velocities of the 2223 phase growth and decomposition were studied, and found to be depended on the precursor sizes. For the 2223 phase growth with decomposition, the optimum sintering time t_{\max} and the maximum amount of 2223 phase were found to be depended upon τ and τ_1 . When the particles size reduces, the optimum sintering time t_{\max} decreases, and the maximum amount of 2223 phase A increases.

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Table 1. 90% of Precursor Particles with the Diameter Less than that of d ; s Expressed a Total Surface Area per Gram Precursor. Time Parameters τ , τ_1 , τ_0 and the Optimum Time t_{\max} in hours, and the Maximum Amount of 2223 Phase in (x100%)

Precursors	90%< d (μm)	s (m^2/g)	τ (hours)	τ_1 (hours)	τ_0 (hours)	t_{\max} (hours)	$A_{\max} \times 100\%$
A. Freeze-drying	19.6	6.0	22	200	40	51	85
B. Thermal co-decomposition	39.0	1.9	38	220	55	73	79
C. Two-powder	43.0	1.1	70	180	60	89	61
D. Spray-drying	53.0	0.7	160	150	65	152	51

2. MATHEMATIC DESCRIPTION OF Bi-2223 PHASE GROWTH KINETICS

Many researchers have utilized Avrami equation $A = 1 - \exp(-kt^n)$ and combined the Arrhenius equation for the expression of $k = k_0 \exp(-E/RT)$ to determine the activation energy E of 2223 phase [18-24]. The combination equation is in the form of

$$A = 1 - \exp\left[-k_0 t^n \exp\left(-\frac{E}{RT}\right)\right] \quad (1)$$

where A is the relative amount of the 2223 phase in the resultant, t the sintering time, T the absolute temperature of the reaction and R the gas constant; the parameters k_0 and n are reaction constants; and E is the activation energy of the 2223 phase growth, value of which can be fitted from Eq. (1) with experimental data.

It is noted that Equation (1) was derived to describe a simple two-dimensional diffusion-controlled, nucleation-growth thermostatic process. However, the reaction from 2212 phase to 2223 phase is neither a simple two-dimensional diffusion-controlled processing, nor a nucleation-growth one. The 2223 phase growth is a complex three-dimensional process, and does not develop from nucleation, but from giant sheets of 2212 crystals. Furthermore, Equation (1) only describes a monotonic growth. Actually at high sintering temperatures, the 2223 phase growth was observed for a while and then it was decomposed. But equation (1) cannot express a growth with decay process. There are a couple of discrepancies in the Eq. (1) describing bulk 2223 phase growth.

To avoid these discrepancies, Chen *et al.* suggested a different equation to describe the 2223 phase growth [15-17, 26]: $A = 1 - \exp(-t/\tau)$, where t is sintering time, τ a time constant determined by the 2212 phase lifetime. As the grown 2223 phase is not in a stable state and decompose later, the decomposition function will be in the form of $\exp(-t/\tau_1)$, where τ_1 is a time constant determined by the 2223 decomposition. So, at t moment the relative amount of the 2223 phase can be expressed to be

$$A = [1 - \exp(-\frac{t}{\tau})] \exp(-\frac{t}{\tau_1}) \quad (2)$$

The time constants τ and τ_1 in the Eq. (2) are dependent on property of precursor and synthetic conditions. For some particle precursors, the 2223 phase decomposition can be delayed with a period, τ_0 and Eq. (2) becomes to be

$$A = [1 - \exp(-\frac{t}{\tau})] \exp(-\frac{t - \tau_0}{\tau_1}) \quad (3)$$

Equations (2) and (3) can describe growth and decomposition for the 2223 phase. On another hand, the Eq. (1) is not discrepancy with Eqs. (2) and (3), and in some cases, they are compatible. If the 2223 phase does not decompose, the time constant τ_1 tends towards infinite; and if taken $n = 1$ and $\tau^{-1} = k$, Equations (2) and (3) reduce to Eq. (1).

3. EXPERIMENTS AND RESULTS

The starting materials for the Bi-2223 phase were oxides of Bi_2O_3 , PbO , SrCO_3 , CaCO_3 and CuO in the stoichiometry of $\text{Bi}_{1.84}\text{Pb}_{0.35}\text{Sr}_{1.91}\text{Ca}_{2.05}\text{Cu}_{3.05}\text{O}_y$. Precursors of the 2223 phase were prepared through four different routes: freeze drying, thermal co-decomposition, spray drying and the two-powder oxide method, which were described in the refs [3, 4]. The precursors were pressed into pellets using 3 tons pressure and the syntheses of 2223 phase were performed by sintering precursors at 850°C in air for 0, 20, 36, 60, 83, 100, 150 and 197 h, respectively [7-10, 15-18, 26].

The samples were characterized by XRD (Model Philips PW 1730 with $\text{CuK}\alpha$ radiation) and a low-angle laser light scattering (LALLS) device - the Malvern Mastersizer - was used for particle size distribution analysis. The instrument consists of a gas (He-Ne) laser source of coherent light of $0.63 \mu\text{m}$ wave length. It has a high resolution, yielding up to 100 size classes in the range of $0.01\text{-}80 \mu\text{m}$. The determined precursor particle diameters, d and specific surface area, s are showed in the first and second column in Table 1, respectively. The values of d show 90% of precursor particles are with the diameter less than d ; and that of s show a specific surface area, in a total surface area per gram of precursor particles. To perform the XRD experiment, the samples of the 2223 phase were ground in fine powders. Fig. (1) shows XRD patterns of freeze-dried bulk samples sintered at 850°C for various times. The XRD technique was used to estimate the phase composition of the 2223 phase. The calculation formula is in the form of $A = I_{2223}(0010) / [I_{2212}(008) + I_{2223}(0010)]$, where I is the intensity of the XRD peaks [15-18]. It was also employed here to detect the volume fraction of the 2223 phase for different sintering times. Fig. (2) shows the amount of the 2223 phase with four precursors for different sintering times. Time dependences of 2223 phase growth for the four precursors can be described by Eq. (3). Time constants τ , τ_1 and τ_0 in Eq. (3) can be fitted from experimental data and listed in Table 1, as well.

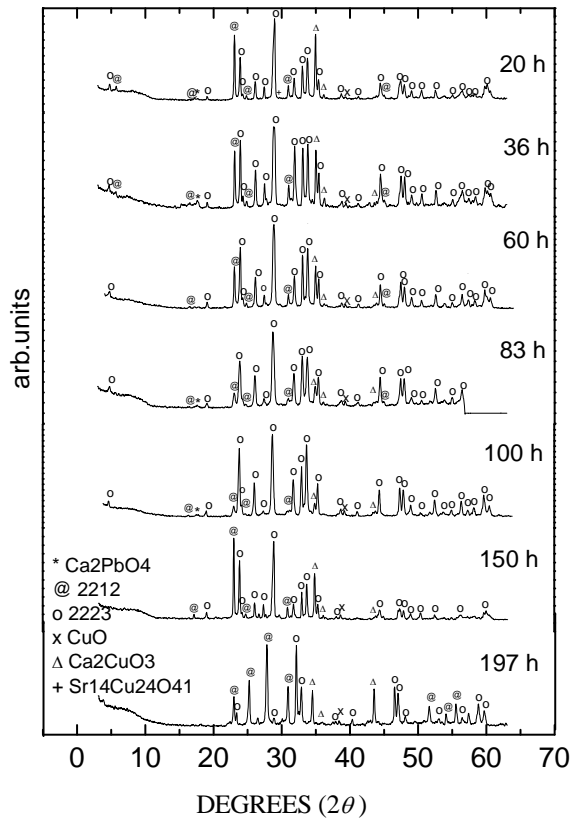


Fig. (1). XRD patterns of freeze-dried bulk samples sintered at 850°C for various times.

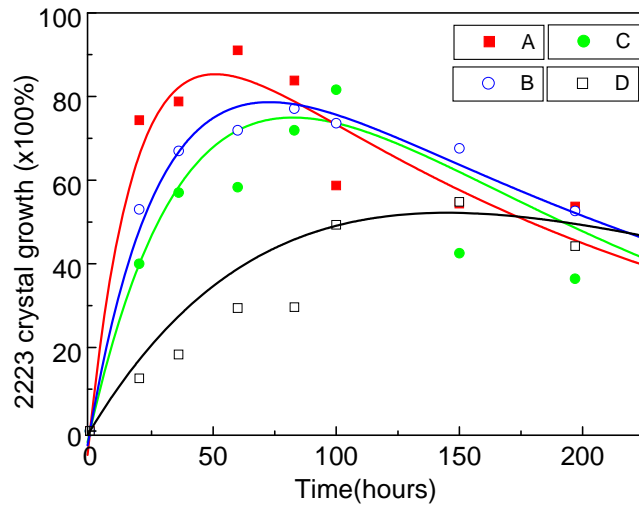


Fig. (2). The amount of 2223 phases as functions of sintering time for the bulk samples prepared from A) freeze-dried, B) thermal decomposition, C) two-powder, and D) spray-dried precursor powders, and described by Eq.(3) in corresponding curves.

4. DISCUSSIONS

4.1. Time Constant τ

It was found that time constant τ in our experiments has changed with the particle sizes of precursors; and we can take the particle diameter, d , or specific surface area, s , as a variable to describe τ . The dependence of τ^{-1} on particle diameter d (μm) is showed in Fig. (3), which can be described by a power-law,

$$\tau^{-1} = c_1 - c_2 d^{1.65} \tag{4}$$

where c_1 and c_2 are constants, fitted to be: $c_1 = 4.55 \times 10^{-2} (\text{h}^{-1})$ and $c_2 = 5.5 \times 10^{-4} (\text{h}^{-1} \cdot \text{d}^{-1.65})$, respectively.

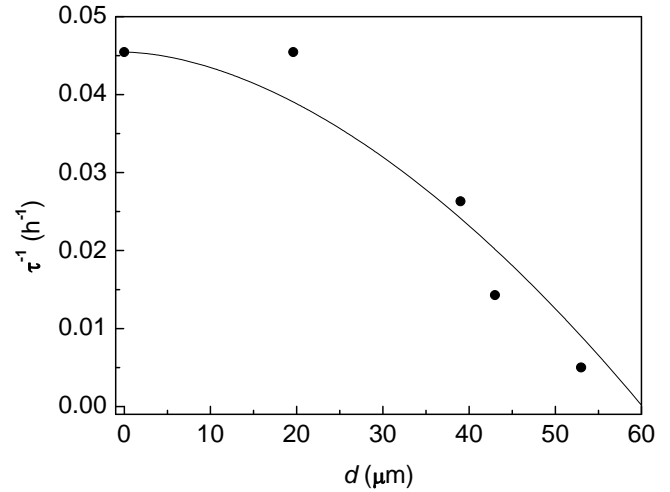


Fig. (3). The τ^{-1} dependence of particle diameter d described by a power-law with an index of 1.65.

Actually, the quantity τ^{-1} possesses own meaning, the maximum velocity of the 2223 growth. With Equation (2), the velocity of the 2223 growth can express to be $dA/dt = \tau^{-1} \exp(-t/\tau)$. At beginning, $t=0$, $dA/dt = \tau^{-1}$ and the 2223 is growing the fastest; and when $t = \infty$, $dA/dt = 0$. The velocity of the 2223 growth is decreased as sintering time t increases, as shown in Fig. (4).

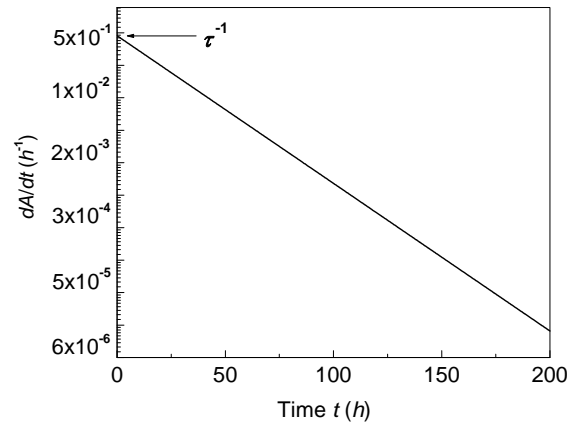


Fig. (4). The velocity of the 2223 growth reduced as sintering time t increasing, and the maximum growth velocity at $t = 0$, with the values of τ^{-1} .

The index of 1.65 in Eq. (4) shows that τ^{-1} depends on the both diameter and surface area of precursor particles. Because, if the τ^{-1} was just dependent on the particle diameters, the index would be 1; and if only dependent on the particle surface area, the index would be 2.

4.2. Time Parameter τ_1

Time parameter τ_1 is the mean lifetime of the 2223 phase, which describes the stability of the 2223 phase at sintering temperature. For the above four precursors and at 850°C sintering temperature, 2223 phase was observed being growth and decomposition. The 2223 phase at that

temperature was in a sub-stable state. The 2223 phase in a sub-stable state can be decomposed back to 2212 phase under some surroundings disturbances. And the decomposition will release the activation energy E . The released energy E can heat the neighbouring 2223 crystals and causes local temperature increase. Due to the local temperature increased, neighboring crystals begin decomposing, and it thus causes releasing the activation energy E , as well. Sequentially the 2223 crystals in the location start decomposing in one after another.

In this experiment, the mean lifetime τ_1 of the 2223 phase is dependent on sintering temperature, i.e. $\tau_1 = \tau_1(T)$. At 850°C, for different precursors the values of τ_1 are in range of $\tau_1 \approx 200 \pm 50$ (h). As a discussion as above, the velocity of the decomposition can be expressed to be: $dA/dt = -\tau_1^{-1} \exp(-t/\tau)$, and the maximum velocity for the composition is τ_1^{-1} .

4.3. Time Parameter τ_0

The time parameter τ_0 describes the delay time of the 2223 growth. Dependence of τ_0 on particle diameter of precursors is shown in Fig. (5). When particle diameter d reduces, value of τ_0 is linearly reducing; and as $d \rightarrow 0$, $\tau_0 \rightarrow 0$. For amorphous precursor, $\tau_0 \approx 0$.

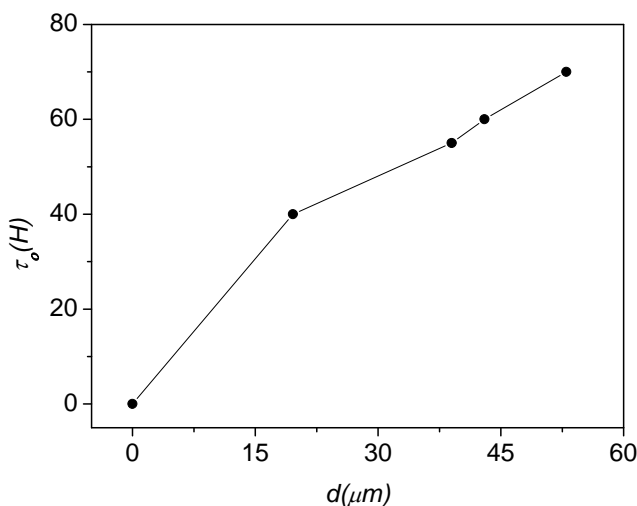


Fig. (5). Value of τ_0 linearly reducing following by particle diameter d .

4.4. The Effects of Specific Surface Area on 2223 Crystal Growth

Large surface area of particles for a certain amount of precursor is an important characteristic, which relates to its chemical activities. Specific surface area (total surface area per gram precursor), s , can show the characteristic. One mole precursor with the stoichiometry of $\text{Bi}_{1.84}\text{Pb}_{0.35}\text{Sr}_{1.91}\text{Ca}_{2.05}\text{Cu}_{3.05}\text{O}_y$, roughly to say, can 'build' one mole 2223 phase, and one mole 2223 phase superconductor is with 6.02×10^{23} unit 2223 crystals. Or say, one unit stoichiometric precursor can 'make' one unit 2223 crystal cell.

To understand the physics meaning of s , we note the value s of precursor A shown in Table 1 is 6.0 in m^2/g , and its corresponding mean diameter is $5\mu\text{m}$. From the view of nano-scale, the unit "m²/g" shall be changed into "nm²/mol";

note the formula weight of the precursor, the transformation can be calculated and written as: $\text{m}^2/\text{g} = 1.04 \times 10^{21} \text{nm}^2/\text{mol}$. And one mole material divided by Avogadro constant N_A , 6.02×10^{23} , will become a unit formula material. The unit "m²/g" can divide by 6.02×10^{23} and become: $\text{m}^2/\text{g} = 1.7 \times 10^{-3} \text{nm}^2/\text{one unit stoichiometric precursor}$. Now the value of $6.0 \text{m}^2/\text{g}$ can be changed to be: $6.0 \text{m}^2/\text{g} = 6 \times (1.7 \times 10^3 \text{nm}^2/\text{one unit stoichiometric precursor}) \approx 0.01 \text{nm}^2$ per unit stoichiometric precursor.

The 2223 crystal lattice constants are $a = b = 0.38 \text{nm}$ and $c = 3.7 \text{nm}$. The surface area per crystal cell, s_{2223} , is around 6nm^2 . For the precursor with diameter of $5\mu\text{m}$, the surface area ratio of per crystal cell to unit precursor's particles is: $s_{2223}/s = 6/0.01 = 600$. The specific surface area of the precursor A is 1/600 as less as that of the 2223 crystal. It means that oxides in the precursor for building the 2223 crystalline are piled in much dense way. Those oxides will absorb energy during diffusion from precursor particles to "build-up" 2223 crystals. That is why the larger particle size the precursor, the longer delay time τ_0 the growth reaction has. Smaller particles size is advantage for the 2223 crystal growth.

The relation of logarithmic s vs d is in good linearity as shown in Fig. (6). When particle size decreases, its s increases and the ratio of s_{2223}/s reduces, as well. If d becomes as small as 50nm , the s_{2223}/s is still as large as 5. When d is less than 8nm , the $s_{2223}/s \approx 1$, the value of s attends to the same dimension that of 2223 cell has. And when the precursor particles had the same dimension with 2223 cells, the density of which become as low as similar to that of 2223 crystal. Certainly, to prepare a precursor with particles in size of the 2223 crystals is attractive. Technically, researchers prefer to prepare amorphous precursor instead of the material with the size less than 8nm .

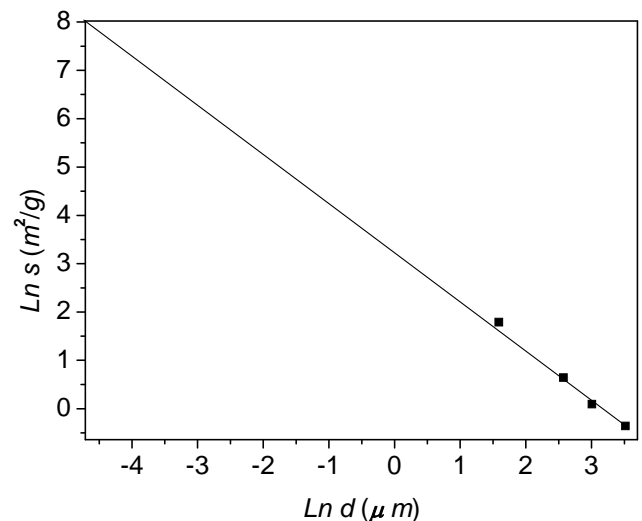


Fig. (6). The relation of logarithmic s and d in good linearity.

4.5. The maximum amount of 2223 composition

The maximum amount of 2223 phase for the four precursor samples can be calculated from the Eq. (3) with the parameters listed in Table 1. With Eq.(3), let $dA/dt = 0$, we obtain

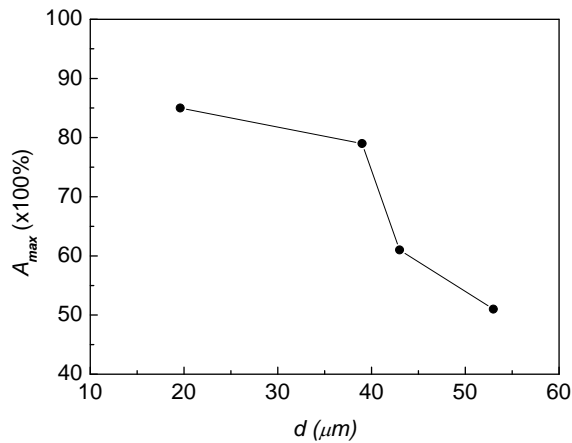


Fig. (7). The maximum amount of 2223 phase increased as the particle diameter of precursor reduced.

$$t_{\text{max}} = \tau \ln \frac{\tau + \tau_1}{\tau} \quad (5)$$

where t_{max} is a optimum sintering time at which the A reaches the maximum. Equation (5) shows that optimum sintering time is only dependent upon the time parameters τ and τ_1 and independent on the delay time parameter τ_0 . The values of t_{max} for the four precursor's samples are changed by particle size, as listed in Table 1.

Substituting Eq. (5) for t in Eq. (3), the maximum amount of 2223 phase can be expressed to be

$$A_{\text{max}} = \frac{\tau_1}{\tau + \tau_1} \left(\frac{\tau}{\tau + \tau_1} \right)^{\tau/\tau_1} \exp\left(\frac{\tau_0}{\tau}\right) \quad (6)$$

where A_{max} is the maximum amount of 2223 phase, which is determined by the three time parameters τ , τ_1 and τ_0 . The

calculated results are listed in Table 1, too. Fig. (7) shows that the maximum amount of 2223 phase A_{max} increases when the particle diameter of precursor d reduces.

5. CONCLUSION

5.1. Kinetic Equations Describing 2223 Phase Growth

The Kinetic Equation (1) was widely utilized to estimate the value of activation energy E , and the estimated values compared with ones obtained from other method were in the same order or to be thought relatively reasonable. There will be serious discrepancies to describe the 2223 phase growth, as discussed in section 2, if the equation (1) is used. First, the growth mechanism of the 2223 phase is inserting oxides into the 2212 sheets. This is quite different from the "simple two-dimensional diffusion-controlled model" based on nucleation-growth processing described by the Eq. (1). Second, the Eq. (1) cannot describe the other properties for the 2223 phase growth, especially for the 2223 phase decomposition. And the phenomena of the 2223 phase decomposition was observed and conformed in our experiment by SEM images, as shown in Fig. (8).

On the other hand, the kinetic equation (2) can describe the 2223 phase growth and decomposition without any difficulties, also can estimate the mean lifetimes for 2212 and 2223 phases and calculate the velocity of 2223 phase growth and decomposition with greater accuracies. But it is not to estimate the activation energy.

5.2. The Effects of Precursors' Sizes on 2223 Crystal Growth

Time constant τ in Eq. (2) or (3) describes the 2212 phase mean lifetime, and τ^{-1} determines the velocity of the 2223 phase growth, which depends on both the precursor particles' diameter and its total surface area. When the

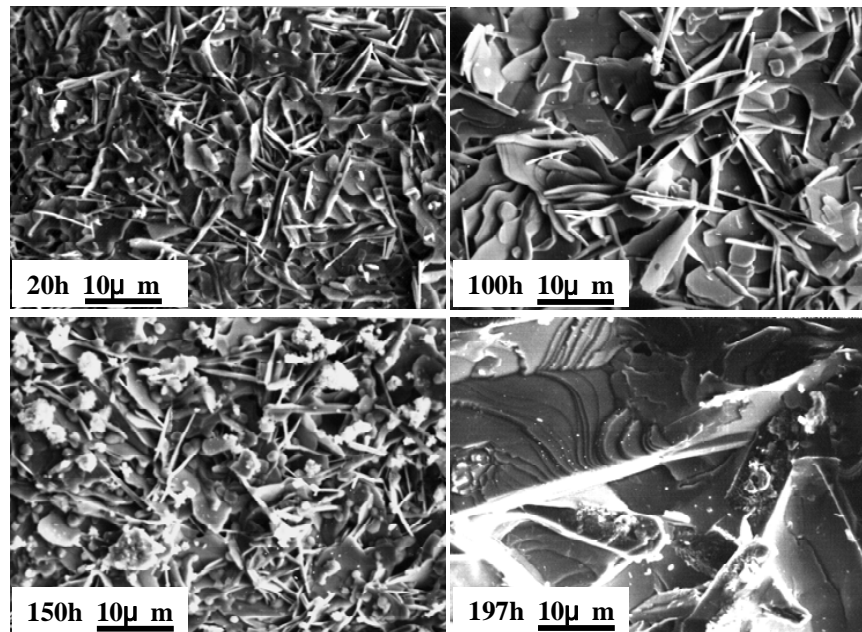


Fig. (8). The 2223 phase composing and decomposing for the thermal co-decomposition samples. Imagine I showed the 2223 crystals appearing after 20 hours sintering; Imagine II showed the maximum of the 2223 phase at 100 hours sintering; Imagine III showed the 2223 phase decomposed after 150 hours sintering; Imagine IV showed the most of the 2223 phase decomposed when sintering 200 hours.

particles size reduces, τ reduces, τ^{-1} increases and the 2223 phase grows fast.

The time constant τ_1 expresses mean lifetime of the grown 2223 phase, τ_1^{-1} the velocity of the 2223 phase decomposition, which is independent on the precursor size.

For the 2223 phase growth with decomposition, the optimum sintering time t_{\max} is dependent on τ and τ_1 , and the maximum amount of 2223 phase depends upon τ , τ_1 and τ_0 . When the particles size decreases, the optimum sintering time t_{\max} decreases, and the maximum amount of 2223 phase A increases.

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