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电子束对Sol-gel法制备的TiO₂的 显微形貌和相变的影响

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簡要:用Sol-gel方法制备了纳米级TiO2,用XRD分析了粉末的相结构和尺寸,并用原位TEM和SED技术详细分析了电子束对不同结构TiO2的影响。 XRD结果表明, 250℃以下干燥的样品为无定形TiO2, 360℃和600℃热处理的样品由锐钛矿相组成, 750℃热处理的样品由锐钛矿(体积分数30%)和金红石(体积分数70%)两相组成, 当温度为950℃时,全部转变为金红石。 在电子束照射下, 除了110℃干燥的团聚体在短时间内未观察到锐钛矿和金红石相外,在250℃和360℃加热的样品中均观察到了金红石相,甚至全部为金红石相。 颗粒尺寸增大, 电子束对相变的影响减弱, 600℃保温1h的样品在电子束照射下短时间内未出现金红石相,然而当样品处于锐钛矿和金红石双相时, 电子束又促进了金红石相的形成, 这与氧缺陷的增加有关。 此外, 在电子束的照射下, 无定形的TiO2样品中还出现了TiC相。

关键字: 电子束; Sol-gel法; Ti0 12; 相变; 显微形貌

Effect of electron beam on microstructures and phase transformation of ${\rm TiO_2}$ prepared by Sol-gel technique

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Abstract: Nano-sized TiO_2 was prepared by Sol-gel technique. The phase and size of the as-derived powders were analyzed by XRD. The effect of electron beam on the microstructures and phase transformation of TiO_2 heat treated at various temperatures for different times was studied by in-situ TEM and SED. It is shown that below 250° C amorphous phase TiO_2 is presented. Upon heating the amorphous phase can be transformed to anatase and rutile and 70% (volume fraction) rutile can be detected after being calcined at 750° C for 1h. The rest was anatase. Given the electron beam, anatase and rutile can be obtained from powders processed at 250° C and 360° C. With increasing sizes, the effect of electron beam on the process of phase transformation is delayed. Rutile phase can not be observed by beam heating for short time in the sample heat treated at 600° C for 1h. However, if the two phases of anatase and rutile coexist, the electron beam can facilitate the transformation to the final stable rutile. These changes may be due to the reactive vacuum atmosphere. In addition, a new phase is identified as TiC after beam heating for some seconds in the sample with amorphous phase.

Key words: electron beam; Sol-gel technique; TiO₂; phase transformation; microstructure

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