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XRD测定炭素材料的石墨化度

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摘 要：研究了用X射线衍射(XRD)测定炭素材料石墨化度的原理和方法,指出其本质是精确测定炭石墨六方晶格的C轴点阵常数值,为此,用高纯硅粉作内标以校准测量误差.为提高测量精度,选用高角度的$C_{(004)}$-$Si_{(311)}$衍射线对;当试样的石墨化度较低时,因$C_{(004)}$衍射线强度太小,选用$C_{(002)}$-$Si_{(331)}$线对.将它们分别进行$K_{\alpha1}$,$K_{\alpha2}$双线分离处理后,以$K_{\alpha1}$峰的半高宽之中心点定峰位并据此计算石墨化度.实验结果表明:在碳/碳复合材料中,由于采用了多种原材料,经高温热处理后形成石墨化的程度不尽相同,即试样中含有不同石墨化度的组分,致使炭石墨的衍射线呈现明显的不对称性,此时必须进行多重峰分离处理,分离出的子峰通常无需再进行双线分离,即可直接用来计算各组分的石墨化度;由各子峰的积分强度可计算不同石墨化度组分的相对含量,以此进行权重计算所得的平均石墨化度更合理地反映了试样内部石墨化度的实际情况.

关键字：XRD;石墨化度;多重峰分离;内标

Graphitization measurement of carbon material by X-ray diffraction

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Abstract: The principle and measurement method for graphitization degree of carbon materials by X-ray diffraction wereinvestigated, through calculating the lattice constantc value of hexagonal systemgraphite precisely. So it was verynecessary to correct measure error using high-pure silicon powder as calibrated standard material. In order to improve measuring accuracy and precision, high-angle diffraction line couple of C(002)-Si(311)should be selected. With regard to the material of low graphitization degree, only diffraction line couple of C(004)-Si(331)could be selected because the diffraction intensity of C(004)was too low. Through separating the diffraction lines to two parts of Kα1and Kα2, graphitization degree was obtained by calculating the half-high-wide center point position of Kα1peak. There exist different graphitization degrees in C/C composite, and so the profile of diffraction pattern was not symmetrical. Somultiple-peak separation should be executed, and the separated peaks could be used to calculate graphitization degree directly. In this condition, there′s no need for the double-peak separation. The integral intensityobtained from every single-peak could calculate the relatecontent of every component, and it was very reasonable to investigate the true graphitization degree of C/C composite.

Key words:XRD; graphitization degree; multiple-peak separation; inter-standard

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