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Combined Differential scanning calorimetry, Raman and Brillouin spectroscopies: a multiscale approach for materials investigation.

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Abstract

A new experimental setup combining DSC, Raman and Brillouin spectroscopies was developed. In order to estimate its accuracy and stability a study of silicon and the alpha-beta quartz phase transition were performed. The data obtained demonstrated good agreement with previous studies using these three different techniques. For quartz, the temperature behavior of its 147 cm^{-1} Raman mode was studied in detail. Using a two-phonon coupling treatment of the Raman band, we show for the first time that its behavior can be well described by Landau theory of first-order phase transitions. The combined DSC-Raman-Brillouin technique is a powerful tool for material science capable of studying thermal, structural and elastic properties simultaneously.

Keywords: Raman spectroscopy; Brillouin spectroscopy; calorimetry; quartz; phase transition

Introduction

Differential scanning calorimetry (DSC), Raman and Brillouin spectroscopies are powerful fundamental tools of modern material science. Information available from a DSC experiment allows for understanding of thermal behavior: phase transitions, curing and other processes occurring in amorphous and crystalline media. These optical spectroscopies are nondestructive methods that make it possible to interpret changes in the material: Raman spectroscopy can provide detailed information about the structure, while Brillouin scattering spectroscopy contains information about its elastic properties.

Raman spectroscopy has additionally been proven as a tool for investigating temperature dependent and phase transition processes. For example it has been applied for *in-situ* monitoring of the curing of epoxy resins[1,2] crystallization processes[3,4], structural changes in glasses under high temperature or pressure and others examples[5–7]. Combined DSC-Raman experimental setups have been implemented[8,9] and have become commercially available[10]. Despite the fact that Brillouin spectroscopy was successfully applied for investigation of order-disorder[11] and liquid-glass[12,13] phase transitions, elasticity measurements at elevated temperatures[14,15] etc., it is still an uncommonly used and quite “exotic” technique for material science.

At the moment all three techniques are commercially available and DSC, Raman and Brillouin measurements could be easily performed separately, e.g. using an additional controlled heating stage. Nevertheless, implementation of a combined experimental setup in which the optical spectroscopies are performed *in-situ* inside the calorimeter should minimize errors in temperature calibration, differences in sample preparation/treatment for a specific experiment, as well as simplifying and accelerating the experimental procedure.

In this study, design of an experimental setup for combined DSC, Raman and Brillouin spectroscopic measurements (also known as **Associated Raman Brillouin Calorimetry**, or simply **ARABICA**), using commercially available components, is described. The temperature stability of the modified DSC and influence of the laser radiation on the sample temperature was checked by calculation of Stokes to anti-Stokes ratio for the first order silicon Raman scattering band. The combined DSC-Raman-Brillouin technique was applied to study the alpha-beta transition of natural quartz. Careful comparison of the results obtained within this work with the literature, was performed to estimate the level of consistency between the experimental methods, as well as the capabilities and limitations of the experimental setup. The high quality of the data acquired for the alpha-beta transition allowed for refining of the parameters at which it occurs. It has previously been shown that changes of physical parameters observed during the transition can be treated using the critical exponent contribution as well as more straight forward Landau theory[16,17]. Here we extend this approach to see if the same behavior can be deduced from the Raman shift observation.

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