Application Note B-XRD1062

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B-XRD1062 - Observation of Lattice Parameter Changes in Silicide Powders Using an Infrared Heating High-temperature Attachment

Introduction

Lattice parameters are constants that define the size and shape of a unit cell, which is the fundamental repeating building block of a crystal structure. They consist of the unit-cell edge lengths (*a*, *b*, and *c*) and the angles between them (*α*, *β*, and *γ*). Lattice parameters can be refined from the peak positions of experimental powder X-ray diffraction profiles. Such refinement makes it possible to investigate subtle changes in the lattice parameters of metals and ceramics under different temperature conditions or with varying solid-solution compositions. The Reactor X high-temperature heating attachment utilizes infrared heating, allowing rapid heating rates. The use of a fused quartz sample holder makes it suitable for the measurement of materials that may react with conventional metal sample holders.

Measurement and results

Figure 1 shows the X-ray diffraction profiles of nickel silicide (NiSi) powder measured at temperatures ranging from room temperature to 1000 °C. As the temperature increases, the diffraction peaks shift either toward lower or higher angles. A shift toward lower diffraction angles indicates an increase in the lattice plane spacing (d-value). Since the direction and magnitude of the peak shifts vary among the reflections, it is expected that the thermal expansion coefficients of NiSi lattice parameters a, b, and c differ from one another, and that one of these coefficients may be negative.

Figure 2 shows the lattice parameters *a*, *b*, and *c*, determined by the Whole Powder Pattern Fitting (WPPF) method, as a function of measurement temperature. The angles *α*, *β*, and *γ* were fixed at 90° by symmetry. This result revealed that the thermal expansion of *a*, *b*, and *c* is anisotropic, and that the thermal expansion coefficient of b is negative. It also showed that, although the thermal expansion exhibited a gradual nonlinear dependence on temperature, the unit-cell volume increased linearly.

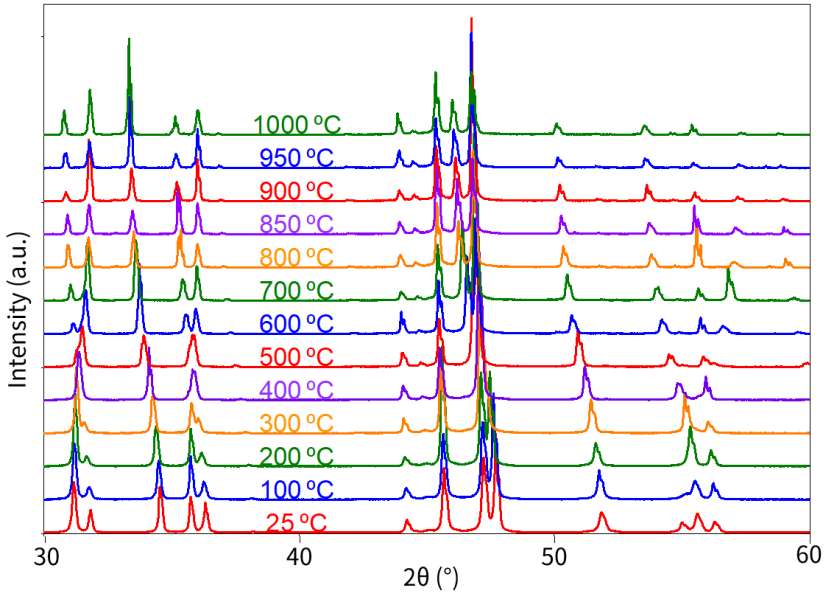

Fig.1: XRD profiles of NiSi powder measured at each temperature

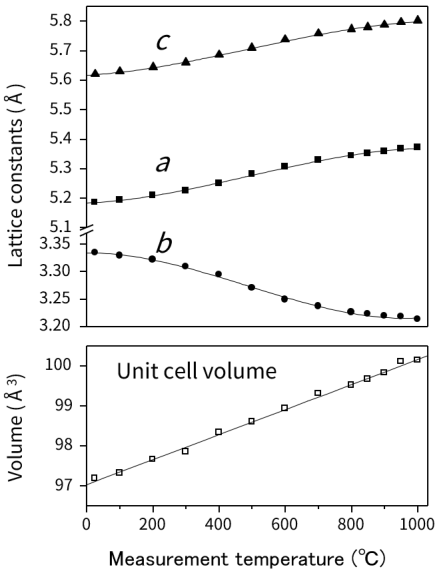

Fig.2: Temperature dependence of the lattice parameters *a*, *b*, and *c*, and the unit-cell volume of NiSi

Recommended equipment and software

- SmartLab automated multipurpose X-ray diffractometer + Reactor X high-temperature attachment for reactive gases
- SmartLab Studio II integrated X-ray analysis software (Powder XRD plugin)

Related products

SmartLab

Advanced, state-of-the-art, high-resolution XRD system powered by Guidance expert system software

SmartLab SE

Highly versatile multipurpose X-ray diffractometer with built-in intelligent guidance

SmartLab Studio II

Windows-based software suite for Rigaku's X-ray diffractometers