# The elastic constants of FCC Ni–Ga and Ni–Ge alloys up to 1100 K

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## Abstract

The elastic constants, $C_{ij}$, of monocrystalline Ni–12.16Ga and Ni–11.59Ge (at.%) were measured to 1100 K. $C_{44}$ is roughly equal for both solid solutions and $Ni_3Ga$ and $Ni_3Ge$. Otherwise, $Ni_3Ga$ is elastically softer, while $Ni_3Ge$ is elastically harder above 900 K. Implications of this latter comparison are discussed.
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## 1. Introduction

Nickel-base superalloys consist of a face-centered cubic nickel-rich solid solution matrix-phase, $\gamma$, within which coherent particles of the ordered intermetallic compound $\gamma'$ are dispersed. The $\gamma'$ phase, which provides the high strength and unique creep resistance of these materials, has the ordered $Cu_3Au$ ($L1_2$) crystal structure. In real superalloys the chemistries of both the $\gamma$ and $\gamma'$ phases can be complex and, moreover, can change as a function of temperature, because the equilibrium compositions of both phases are temperature dependent. Since the lattice constants and elastic constants are dependent on temperature and composition, the lattice mismatch, $\varepsilon$, and elastic-modulus mismatch, $\Delta C$, between the two phases can also be expected to vary with temperature in any given alloy, in turn affecting both microstructural evolution and the kinetics of coarsening of $\gamma'$ precipitates [1–3]. The dependencies of $\varepsilon$ and $\Delta C$ on temperature and composition is not easy to measure in superalloys. However, a handful of binary alloys exist from which useful information on $\varepsilon$ and $\Delta C$ can be measured. In principle, therefore, a great deal can be learned from investigations on binary alloys about the roles these parameters play in microstructural evolution in superalloys.

To date, experimental data on the temperature and composition dependencies of the elastic constants are available only for Ni–Al and Ni–Si alloys [4–7], but the solutes Ga, Ge and Ti have phase diagrams similar to that of Ni–Al in that precipitates of $Ni_3X$ (X = Ga, Ge, Si, Ti) with the $L1_2$ crystal structure form on aging supersaturated Ni–X solid solutions. These binary alloys provide different combinations of $\varepsilon$ and $\Delta C$, hence accurate measurements of their elastic constants could provide valuable insight into the underlying physics of coarsening and microstructural evolution, not only under stress-free conditions, but also under the influence of an applied stress.

The elastic-stiffness constants of the ordered intermetallic compounds $Ni_3Ga$ and $Ni_3Ge$ have been measured over the temperature range 77–1200 K by Tanaka et al. [8] using the rectangular parallelepiped resonance (RPR) method. The main purpose of the present work was to measure the single-crystal elastic constants of Ni–Ge and Ni–Ga solid solution alloys as a function of temperature.

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## 2. Experimental procedures

### 2.1. Specimen preparation

A single-crystal of a Ni–Ga alloy approximately 25 mm in diameter and 80 mm long, grown by the Bridgman method, was purchased from the Alloy Preparation Facility of the Ames Laboratory (Ames, IA). The crystal was annealed in a vacuum furnace at 1423 K for 72 h for the purpose of homogenizing it, but the composition gradient along its length was large and homogenization at the length scale of the crystal was not possible. The Ni–Ge alloy was prepared by arc-melting several buttons from high-purity starting materials. Small pieces cut from the buttons were placed in a small, flat-bottomed alumina crucible, melted inside a vacuum furnace under a pressure of $10^{-3}$ Pa and cooled slowly (3 K/h) through the melting range, yielding a bulk polycrystalline sample with a few large grains exceeding 10 mm in size. Single-crystal specimens were oriented and cut from the larger grains. Additional details of the sample preparation procedures are described elsewhere [9].

For the RPR experiments we used specimens shaped as right-rectangular parallelepipeds with faces normal to {100}. Specimens were cut from the single-crystals using a low-speed diamond wheel after orienting the crystals using Laue X-ray back-reflection. The faces of the parallelepiped specimens were mechanically polished and perpendicularity achieved by carefully mounting them on a polishing jig along with a precision-machined hardened steel "reference" cube. A final check of the orientation showed that deviation of each face from {100} did not exceed 2°. The specimens were solution treated in an Ar atmosphere at 1423 K for 0.5 h, followed by quenching into refrigerated brine (274 K). The faces of specimens were metallographically polished to bring the dimensions of the samples to their final sizes reported in Table 1. The small differences in the dimensions of the three sides of the parallelepipeds (about 50 μm, Table 1) were deliberately introduced to eliminate degeneracies in the resonant frequency spectra that occur when the length of each edge is identical. Eliminating the degeneracies, while at the same time ensuring that the resonant frequencies of similar mode types are close to each other, is helpful when attempting to identify the mode type resonating at a particular frequency [5].

The composition of each sample was determined using energy-dispersive X-ray spectroscopy; the Ni–Ga alloy contained 12.16 ± 0.23 at.%Ga and the Ni–Ge alloy con-

tained 11.59 ± 0.21 at.%Ge. The composition of the Ni–Ga alloy is in the single-phase solid-solution region of the phase diagram over the entire temperature range of the experiment, but the Ni–Ge alloy is not, since the solvus temperature for a Ni–11.59%Ge alloy is ∼950 K. We estimate that the volume fraction of the Ni$_3$Ge in the Ni–Ge sample is about 12% at 773 K [10], below which decomposition is extremely slow. The densities of the specimens were measured by the immersion method and also calculated using values of the lattice parameters at room temperature [11]. These data are shown in Table 1. The measured densities were used for determining the elastic constants from the measured resonant spectra.

### 2.2. Measurement of the elastic-stiffness constants

In the RPR technique, $C_{11}$, $C_{12}$ and $C_{44}$ of an oriented single-crystal specimen are determined from measurements of its mechanical resonance spectrum using information on its dimensions and density. There is no analytical method for solving the inverse problem of deducing the elastic moduli from the measured spectrum, thus an indirect method is used. A starting set of resonant frequencies is calculated using estimated values of the $C_{ij}$ (calculated theoretically or taken from the literature). The difference between the calculated and measured resonant frequency spectrum is minimized, in a least-squares sense, by an iterative process until a "best-fit" set of elastic constants is obtained. Redundancy checks are possible, in principle, by observing more resonant modes than the number of independent $C_{ij}$ for the material. A description of the RPR method, including its application at high temperature, is given by Prikhodko et al. [7]. More thorough reviews are provided by Maynard [12], Migliori and Sarrao [13], Leisure and Willis [14], and Schwarz and Vuorinen [15].

We measured and identified 16 and 40 modes, respectively, for the Ni–12.16%Ga and Ni–11.59%Ge specimens from room temperature to 1100 K in increments of 25°. Temperature measurements were made with two (sometimes three) Pt vs. Pt–13%Rh thermocouples. Uncertainty in the temperature across the dimensions of the specimen was about 5° at the highest temperature reached. The specimens were protected from oxidation during heating by flowing Ar gas. We made several temperature excursions for each specimen (Ni–12.16%Ga: RT to 600 K twice and RT to 1100 K once; Ni–11.59%Ge: RT to 600 K twice and RT to 1100 K twice) and found excellent agreement in the temperature dependencies of the modal frequencies when comparing the results from different runs.

Since the specimens were held between alumina buffer rods (Goto and Anderson [16]) instead of being in direct contact with the transducers during heating, some modes seen at room temperature were not observed during the high temperature runs. Only the modal frequencies measured over the entire temperature range for each specimen were used to reduce the data. In other words, the determi-

Table 1
Composition, dimensions, and densities of specimens used in measurements of elastic constants

| Composition (at.%) | Edge dimensions (mm) | Density (Mg/m³) | |
|---|---|---|---|
| | | Calculated | Measured |
| 12.16 Ga | 5.231 × 5.155 × 5.081 | 8.899 | 8.901 ± 0.014 |
| 11.59 Ge | 1.940 × 1.920 × 1.850 | 8.984 | 8.940 ± 0.040 |