The sensing diode was mounted inside and at the end of this tube, and could be positioned within 1 cm of the crystal without interference with the collimator, as shown in Fig. 2. This tangential mounting produced an airstream of about 1 cm diameter at the capillary.

To measure the temperature attained inside the capillary, a small (less than 0.5 mm diameter) unmounted bead thermistor was inserted in the capillary, and calibrated as above. No detectable temperature difference was found between the temperature measured at the tip of the exit tube and the temperature measured on the near wall of the capillary, with set temperature equal to 4°C. The lowest temperature that could be produced inside the capillary was 15°C, with an airflow of 101 min⁻¹ and cooling water at 20°C.

It was found that temperature gradients within the cross section of the airstream were negligible, but a gradient of 6°C existed across the 1 mm diameter of the capillary. However, by careful alignment of the crystal in the capillary, the effect of the gradient could be minimized, and large-angle precession photographs taken without the large capillary motion affecting the airstream.

Positioning of the exit tube co-axially with the capillary is also possible and should eliminate temperature gradients (Marsh & Petsko, 1973). The design of the exit tube in this case may require the addition of a Mylar extension (Hovmöller, 1981) to avoid deflection of the laminar flow by external air currents. This problem was not observed with the airstream tangential to the capillary.

The unit was designed to operate at 4°C and uses a supply of dry air, hence condensation of water on the outside capillary surfaces has not been a problem. It was found that condensation of mother liquor inside the capillary could occur at this temperature with the unit mounted tangentially. This problem could be partly overcome by wet mounting the crystal in a minimum of mother liquor and omitting mother liquor plugs along the length of the capillary.

This cooler provides an economical and reliable means of reducing radiation damage and prolonging crystal life, and its compact design will allow remote crystal cooling for most applications.

References


Thermal expansion coefficients of a binary alloy Ni₈₀Zr₂₀ at elevated temperatures. By S. K. SHADANGI, Department of Physics, Government Post-graduate College, Durg (MP), India, U.K. SHADANGI, Research and Control Laboratory, Bhilai Steel Project, Bhilai (MP), India and S. C. PANDA, Department of Physics, Government College of Engineering and Technology, Raipur-492002 (MP), India

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Abstract

The Debye–Scherrer pattern of the alloy Ni₈₀Zr₂₀ clearly shows the presence of a nickel solid-solution phase along with a new intermetallic phase Ni₃Zr₆, which seems to be isostructural with the Co₃Zr₆ phase. The thermal expansion coefficient of the Ni₃Zr₆ phase has been investigated in the temperature range 1003–1493 K. Linear variation of lattice parameter with temperature has been observed. The thermal expansion coefficient remains almost constant throughout this temperature interval.

Measurement of the thermal expansion coefficient of intermetallic compounds has attracted the attention of many investigators. However, there have been relatively few reports on the thermal expansion of intermetallic compounds of the Ni–Zr binary system (Shadangi, Panda & Bhan, 1982, 1983). The present study forms part of our research programme.

Reactor grade Zr and 99-99% pure Ni (Johnson Matthey & Co. Ltd, London) were used for the preparation of an alloy Ni₈₀Zr₂₀. The alloy was melted at Bhabha Atomic Research Centre, Trombay, in a non-consumable electrode argon arc furnace. The alloy was inverted and remelted several times to promote homogeneity. The weight change after melting was less than 0.1% of the total alloy weight. Therefore no chemical analysis of the alloy was considered necessary. Powders obtained after filing the ingot were sieved through a 325 mesh screen and were sealed under vacuum in silica capsules. These capsules were heated to
different temperatures and were rapidly quenched in water so as to arrest the phase corresponding to the particular temperature. The temperature controller of the resistance furnace had previously been calibrated for the whole range of temperatures at which the powders were annealed. Debye–Scherrer photographs were taken with Cu Kα radiation (filtered) and an exposure of about 10 h was found sufficient to bring out the details of the photographs. To achieve better accuracy each X-ray photograph was followed by duplicate runs and line positions were measured two or three times.

All the X-ray patterns of this alloy exhibited the presence of the nickel solid-solution phase, Ni(Zr), along with the lines of a new phase. All the lines of this new phase could be very well indexed with a f.c.c. structure (Shadangi & Panda, 1982), which is closely related to the Co23Zr6 phase. As cobalt and nickel both belong to group VIII of the Periodic Table, their alloying behaviour with zirconium will be very similar (Panda & Bhan, 1973). Hence, the new phase, Ni23Zr6, at this composition seems to be isostructural with the Mn23Th6-type structure. This new phase could not be isolated at this composition.

Lattice-parameter values were determined from the line positions of the intense reflections 640, 800, 733, 822, 751, 884 and 10.6.6. The method suggested by Taylor & Sinclair (1945) and Nelson & Riley (1945) was used to determine an accurate value of the lattice constant at each temperature by plotting lattice-parameter values against $\frac{1}{\cos 2\theta/\sin \theta + \cos^2 \theta/\theta}$ and then extrapolating the function to $\theta = 90^\circ$.

The standard deviation (s.d.) in the lattice constant so obtained was calculated for each temperature and it has been observed that the s.d.'s are almost of the same order of magnitude (Table 1). The lattice parameter is found to vary linearly with temperature (Fig. 1), which is expressed by the following analytical relation;

$$a_T = a_1 + a_2 T,$$

where the numerals within the brackets show the s.d. of the constants of the linear equation and $a_T$ is the lattice parameter at $T$ K.

The linear coefficient of thermal expansion, $\alpha = \frac{1}{a} \times \frac{da}{dT}$, at each temperature was evaluated along with their s.d.'s and are listed in Table 1.

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References


Table 1. Lattice parameter and thermal expansion data of the Ni23Zr6 phase at different temperatures

<table>
<thead>
<tr>
<th>Heat treatment and water quenching</th>
<th>a parameter (Å)</th>
<th>Coefficient of thermal expansion (K–1)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Observed</td>
<td>Calculated from linear equation</td>
</tr>
<tr>
<td>Temperature (K)</td>
<td>Time (min)</td>
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</tr>
<tr>
<td>298 (As cast)</td>
<td>—</td>
<td>11.3534(37)</td>
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<tr>
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</tr>
<tr>
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<td>50</td>
<td>11.5324(37)</td>
</tr>
</tbody>
</table>

Fig. 1. Plot of lattice parameter, $a$, versus temperature, $T$, for the alloy Ni30Zr20.