HITACHI SCIENTIFIC INSTRUMENT
TECHNICAL DATA STEM

SHEET NO. 3

SUBJECT: APPLICATIONS OF A NEW MICRO-SAMPLING TECHNIQUE FOR METAL SPECIMEN PREPARATION

INSTRUMENT: FB-2000A FOCUSED ION BEAM SYSTEM
HD-2000 ULTRA-THIN FILM EVALUATION SYSTEM

CONTENTS:

1. INTRODUCTION

An FIB (focused ion beam) micro-sampling technique has been used for specimen preparation for TEM microscopy of semiconductor devices and other electronic materials as it allows direct and site-specific specimen preparation from a bulky sample. This technique is also useful for metal specimens since they are often deformed by mechanical forces applied during the initial cutout in the specimen preparation. We have applied this technique for preparation of Ni-alloy material. We report on the specimen preparation procedures and microscopy results.

2. A NEW MICRO-SAMPLING ATTACHMENT AND ITS OPERATION

The micro-sampling attachment has been developed for use on the FB-2000A focused ion beam system. It allows initial specimen cutout of the area of interest and thinning the specific area of interest within the specimen chamber of the FB-2000A. The thinned specimen section is mounted on an FIB/TEM compatible holder for microscopy. Fig. 1 shows a flow of specimen preparation procedures using the micro-sampling attachment. A sample is mechanically cut out at a size of about 4 mm × 4 mm × 1 mm and is mounted on a bulk specimen holder. The specimen is ion milled around the area of interest and a trench is made (1). The specimen is then tilted at about 60° and the bottom is cut (2). Next, a micro-probe is bonded on the micro-sample using a tungsten deposition for picking up the micro-sample (3). The micro-sample is transported to an edge of a TEM specimen-carrier and bonded on it using a tungsten deposition (4) and thinned (5) for microscopy.

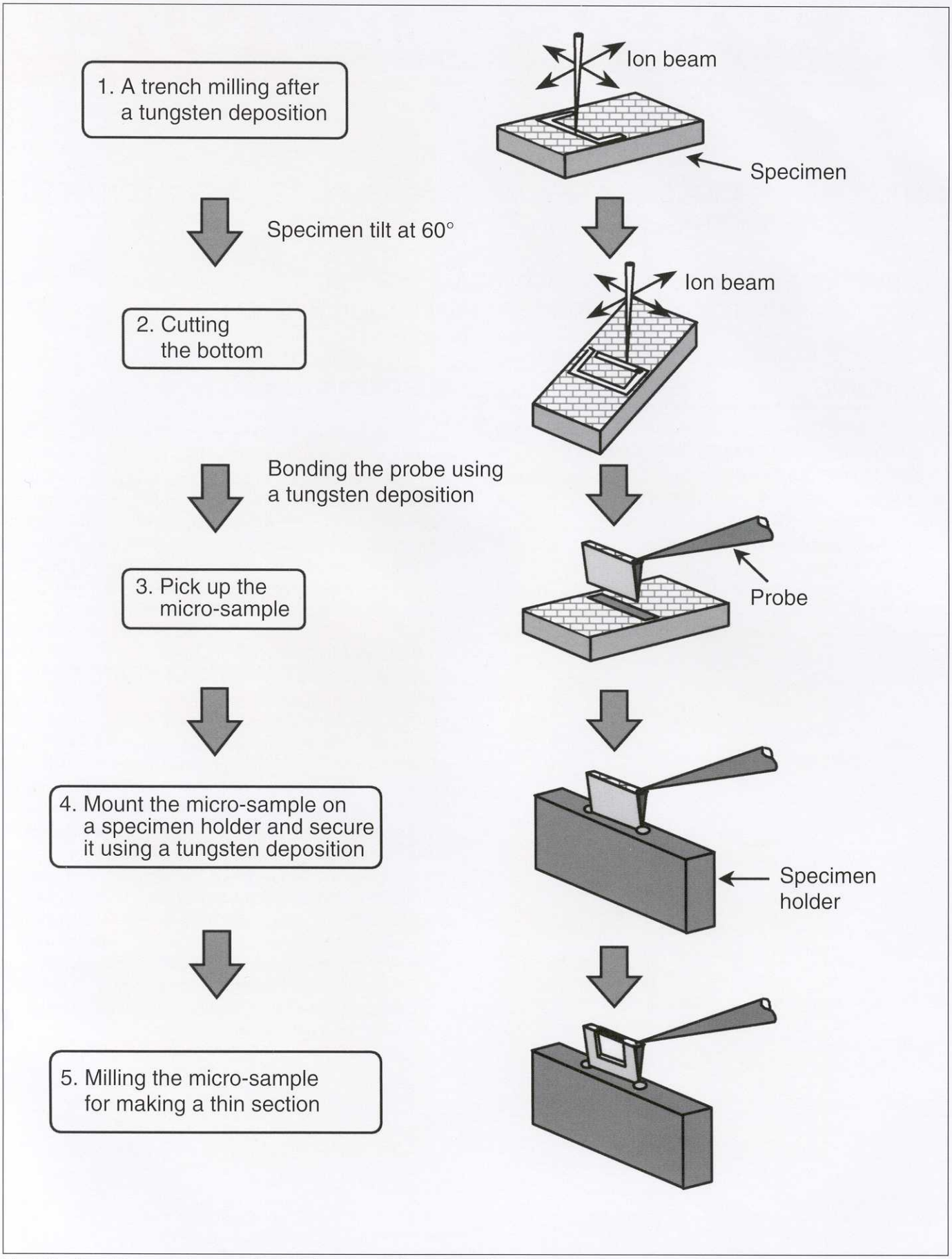

Fig. 1 The flow of the new micro-sampling technique for specimen preparation

3. SPECIMEN PREPARATION AND TYPICAL EXAMPLES

A specimen of Ni-based single crystal super alloy including Re was selected. It has been studied as one of the promising high temperature alloys. We used a specimen which was creep tested at 900°C and 40 kgf/mm^2 for 20 hours. Fig. 2 shows SIM (Scanning Ion Microscopy) images taken during the micro-sample preparation. After a tungsten deposition on the surface of the specimen (1), a step-milling (for cutting the bottom of the specimen) and a trench-milling are performed leaving a micro-bridge which supports the micro-sample (2). The specimen is then tilted at 60° and the bottom is cut (3). After bonding the probe on the micro-sample using a tungsten deposition (4), cut the micro-bridge (5) and pick up the micro-sample (6). The micro-sample is transported to an edge of a TEM specimen-carrier and is bonded on it using a tungsten deposition (7). The probe is cut using an FIB (8). A series of micro-sampling procedures has been completed at this point (9). The micro-sample is then milled to a thin section using a normal milling technique of the FIB for TEM microscopy.

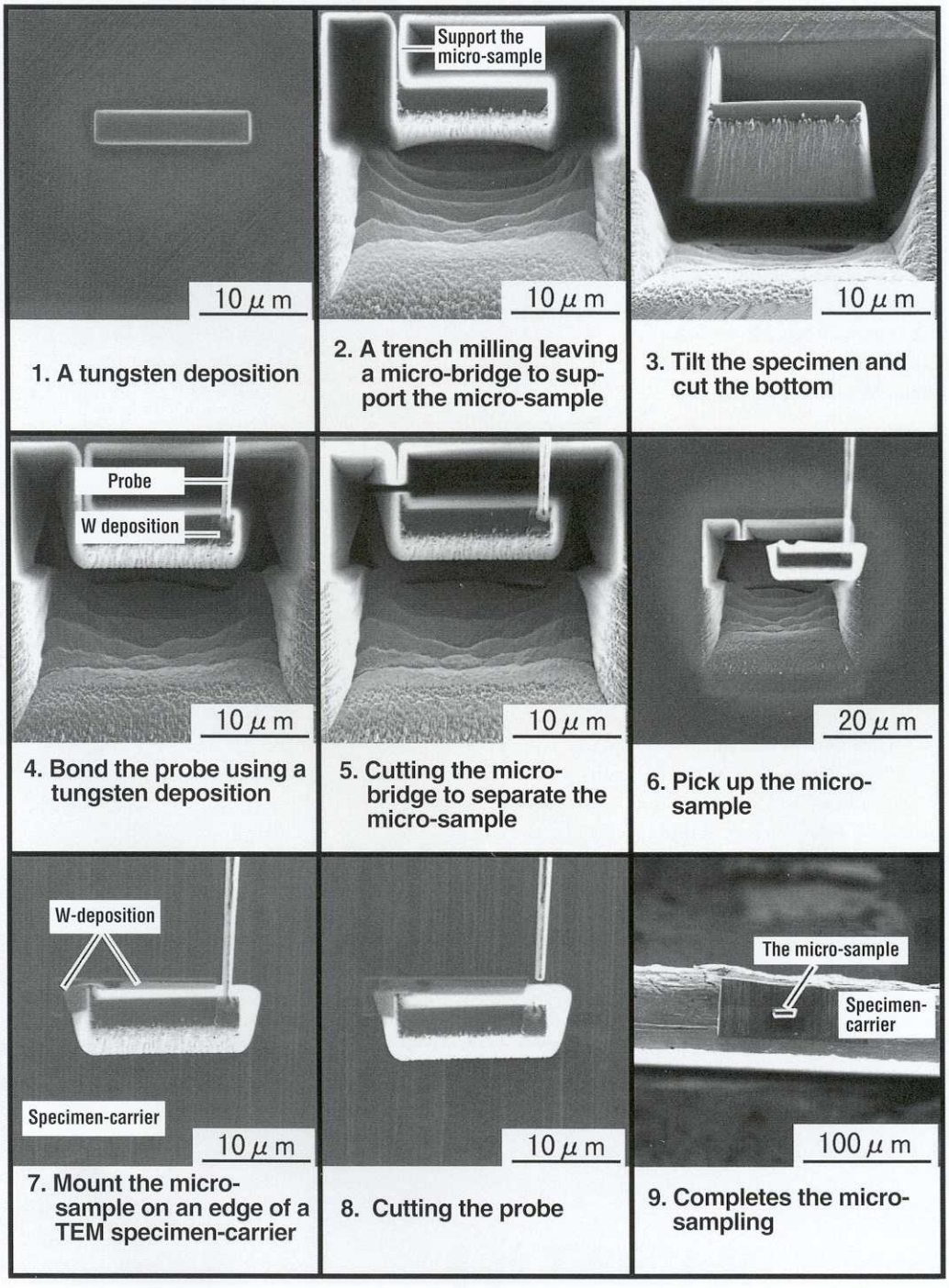

Fig. 2 SIM images showing the micro-sampling process using a Ni-based single crystal super alloy specimen

4. MICROSCOPY AND ANALYSIS

4.1 STEM image

Fig. 3 shows a dark field STEM image (or Z-contrast image) of Ni-based single crystal super alloy prepared by the micro-sampling technique. For microscopy, we have used the HD-2000 ultra-thin film evaluation system. The operating voltage is 200 kV and the magnification is ×50,000. The cross-section shows a net structure called a raft structure. Z-contrast images generally show the differences of atomic numbers and densities of materials at higher contrast than those available with normal bright field images. The lattice phase is a so called γ-phase and the phase surrounded by γ-phase is called γ′-phase. This phase appears when the sample is uniformly heat-processed and aged. It is reported that the net structure is formed in a single crystal alloy processed as mentioned above.

4.2 X-ray mapping images

Fig. 4 shows typical x-ray mapping images of Ni-based single crystal super alloy specimen prepared using the micro-sampling technique.

These images were recorded using the HD-2000 with Noran's Vantage EDX spectrometer. The probe size was about 1 nm and acquisition time was about 8 minutes. We examined 5 elements of Ni, Al, Re, Cr, and Co. Added elements such as Re, Cr, and Co were distributed in γ-phase, and Ni and Al were found in γ′-phase.

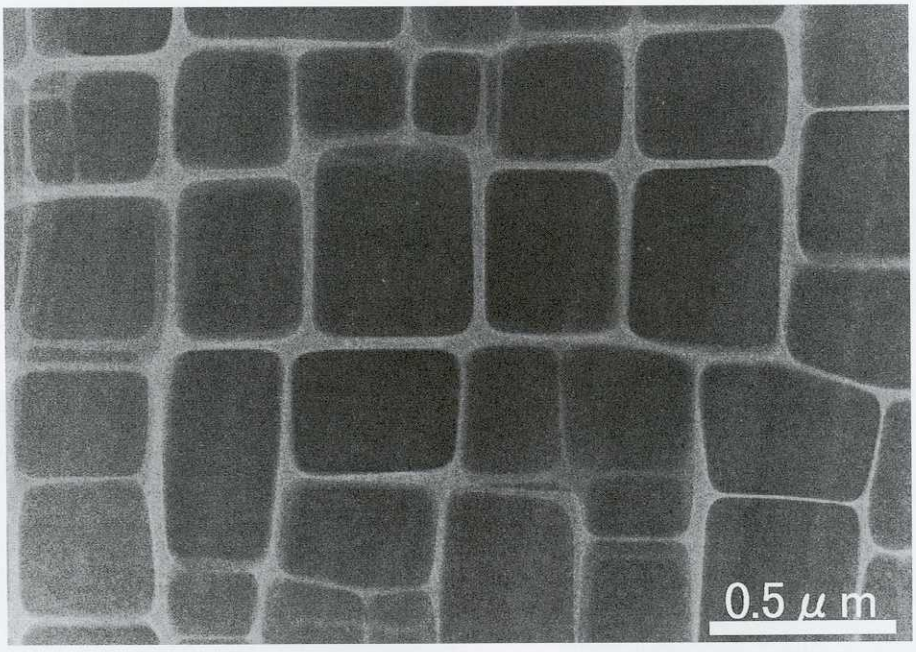

Fig. 3 A dark field STEM image of a Ni-based single crystal super alloy specimen prepared using the micro-sampling attachment
Instrument: HD-2000 ultra-thin film evaluation system
Accelerating voltage: 200 kV
Magnification: ×50,000

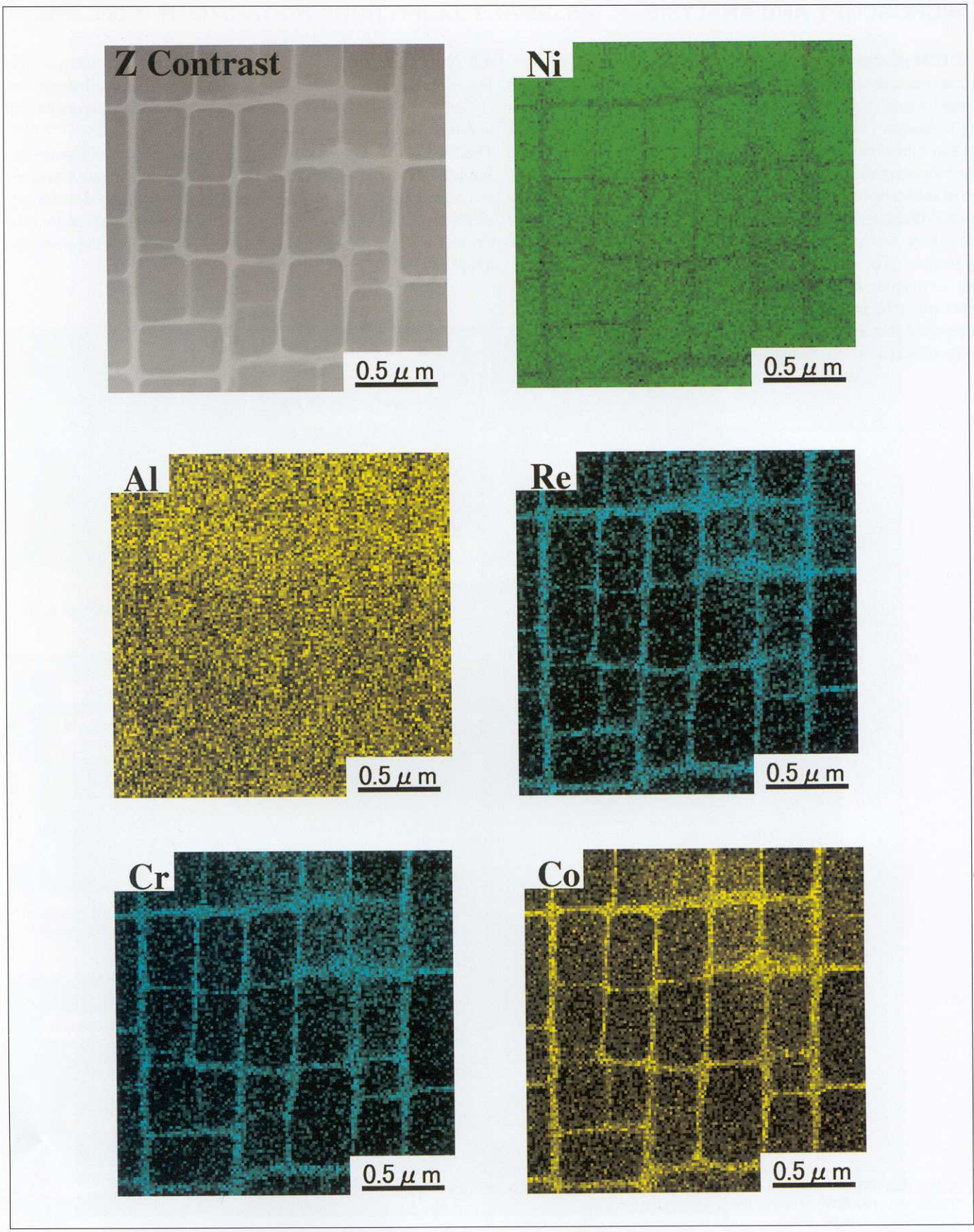

Fig. 4 X-ray mapping images of a Ni-based single crystal super alloy specimen prepared using the micro-sampling attachment
Instrument: HD-2000 ultra-thin film evaluation system
Accelerating voltage: 200 kV
Acquisition time: About 8 minutes
EDX system: Noran Vantage

5. CLOSING REMARKS

We have introduced a new micro-sampling attachment and typical applications using a Ni-based single crystal super alloy specimen. The micro-sampling attachment allows TEM specimen preparation from bulky samples quickly and it will be found useful for semiconductors, metals, ceramics and many other advanced materials. It also allows preparation of multiple specimens, each with different orientations, from a selected small area of interest so that it permits study of the same specimen at various view points. Finally, we would like to thank Drs. H. Harada and M. Osawa, National Research Institute for Metals, Japan for providing the precious specimens and for giving helpful guidance for our study of material structures.

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For further information, please contact your nearest sales representative.

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