

# Application News

## No. P105

### Electron Probe Microanalyzer

### Analysis of Titanium Materials

Titanium materials have mechanical properties such as corrosion resistance and heat resistance and are used in a variety of fields ranging from aerospace to consumer products. For example, general-purpose materials are used in liquefied fuel tanks, aircraft engines, bicycle gears, and other applications, while special materials are used in eyeglass frames and orthodontic wire.

General-purpose materials are classified into three types, unalloyed commercially pure titanium (CP titanium), which is high-purity titanium with excellent corrosion resistance and seawater resistance and is the most general type, corrosion-resistant titanium alloys with excellent corrosion resistance and crevice corrosion resistance, which are secured by adding trace elements to promote the formation of a passive film, and titanium alloys to which alloying elements are added to adjust formability, strength, and other properties. Special materials are broadly divided into high purity titanium, which has higher purity than CP titanium, and functional titanium alloys such as shape-memory alloys and super elastic alloys, which are titanium alloys with additional unique functions.

Titanium materials are utilized in the medical field because of their excellent biocompatibility, and research and development of low-toxicity titanium alloys is being conducted.

This article introduces examples of analysis of a dental root implant made of medical titanium material and an orthodontic wire with super elastic characteristics using an EPMA-8050G EPMA™ electron probe microanalyzer.

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### Analysis of Titanium Alloys

Titanium has an  $\alpha$  phase (hexagonal close-packed: HCP) crystal structure at room temperature and undergoes an allotropic transformation to a  $\beta$  phase (body-centered cubic: BCC) crystal structure at temperatures of 885 °C and higher. Alloys with diverse natures can be produced by adding the  $\alpha$ -phase stabilizing elements such as Al, O, N, and C and  $\beta$ -phase stabilizing elements such as V, Mo, Nb, Fe, Cr, and Ni to adjust its composition. These alloys are classified as  $\alpha$  alloys,  $\alpha$ - $\beta$  alloys, and  $\beta$  alloys.

Ti-5Al-2.5Sn alloy is a representative  $\alpha$  alloy with excellent weldability and high temperature strength (creep characteristics). This alloy also has excellent ductility and toughness at cryogenic temperatures, and is used in liquefied fuel tanks.

As a distinctive feature of the  $\beta$  alloys, because they display the  $\beta$  phase at room temperature, they have outstanding formability in the solution treatment state (before strengthening by heat treatment). Because the highest strength among the titanium alloys is obtained by solution treatment and aging, this type of material is used in golf club heads.

Ti-6Al-4V alloy is a representative  $\alpha$ - $\beta$  alloy, and has a good balance of the features of the  $\alpha$  alloys and  $\beta$  alloys. This alloy, also called 6-4 titanium, has high formability and weldability, and high strength can be achieved by heat treatment. It is used in a wide range of products from aircraft to consumer products.

A dental root implant made of Ti-6Al-4V alloy was analyzed by EPMA, and the dispersion states of the  $\alpha$ -phase stabilizer Al and  $\beta$ -phase stabilizers V and Fe were confirmed, as shown in Fig. 1.

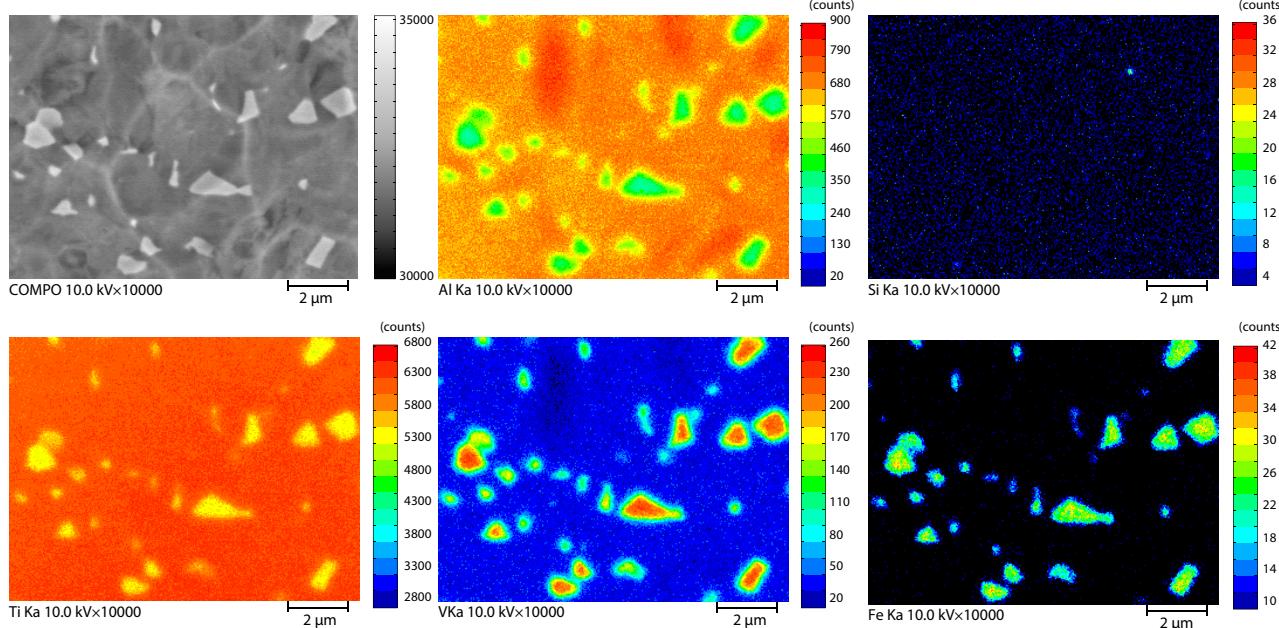


Fig. 1 Analysis of Dental Root Implant

## ■ Super Elastic Alloys

The alloy of Ti and Ni with a 50% ratio of each element is called TiNi alloy (Nitinol), and is known as an alloy with outstanding shape-memory and super elastic characteristics. Shape-memory alloys return to their original shape before deformation if heated to their transformation temperature (transformation point) or higher after deformation to an arbitrary shape, while super elastic alloys have the feature of deforming when a load is applied, but then returning to their original shape when the load is removed. The transformation temperature can be changed by varying the mixing ratio of Ti and Ni, and properties can also be changed by addition of additive elements.

Fig. 2 shows the result of a mapping analysis of an orthodontic wire made of a super elastic alloy. An O, Ti-enriched compound phase with a size of several  $\mu\text{m}$  can be observed as a dispersion distributed microstructure in the TiNi alloy parent phase.

Fig. 3 shows the result of a mapping analysis in which the compound phase was enlarged. Fine spherical particles with about 300 nm diameter can be observed. The concentrations of Ti and Ni in the TiNi alloy parent phase are approximately 46 wt% and 54 wt%, respectively, and their atomic ratio is almost 1 : 1. The concentrations of O, Ti, and Ni in the compound phase are approximately 4.6 wt%, 59.7 wt%, and 35.7 wt%, respectively, and their atomic ratio is about 1 : 4 : 2.

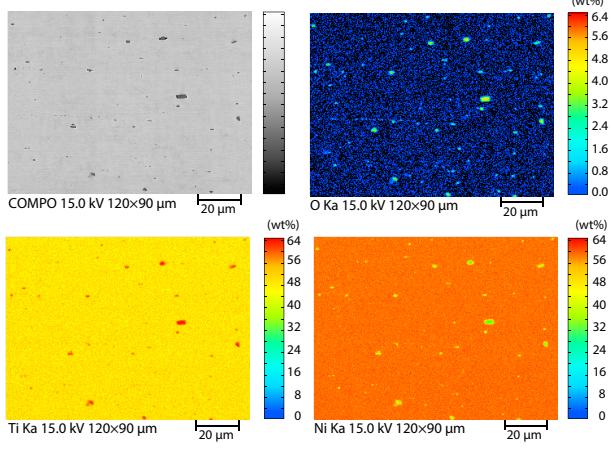


Fig. 2 Analysis of Orthodontic Wire

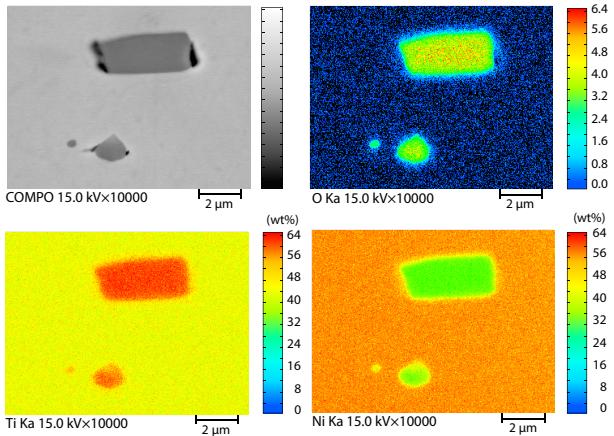


Fig. 3 TiNi Alloy and Ti<sub>4</sub>Ni<sub>2</sub>O Compound Phase

## ■ Phase Analysis of Compound Phase

A phase analysis can show a compound phase accurately because the intensities (concentrations) plotted in the scatter diagram are extracted as point-sets (clusters). In the 3-element scatter diagram of O, Ti, and Ni in Fig. 4, in which phase analysis treatment was

applied to the mapping analysis data for O, Ti, and Ni in Fig. 3, two clusters can be observed corresponding to the position showing the Ti<sub>4</sub>Ni<sub>2</sub>O compound (O, Ti, Ni : 4, 59, 37 wt%, 13, 58, 29 at%) and the cluster center. The phase diagram in Fig. 5, which is separated into the TiNi parent phase and the Ti<sub>4</sub>Ni<sub>2</sub>O compound phase, can be obtained by filter treatment of these respective clusters.

The multi-element scatter diagram in Fig. 6 shows the result of compound display of the TiNi alloy parent phase and the Ti<sub>4</sub>Ni<sub>2</sub>O compound. Here, it can be confirmed that the positions of the compounds coincide with the center of the clusters in the respective 2-element scatter diagrams for O, Ti, and Ni.

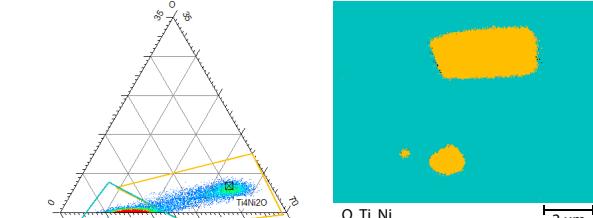


Fig. 4 3-Element Scatter Diagram

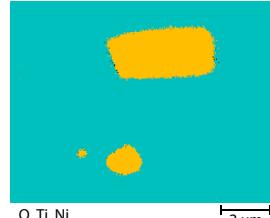


Fig. 5 Phase Diagram

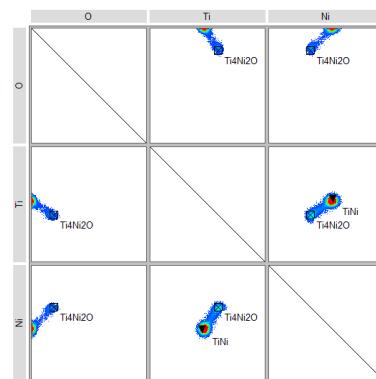


Fig. 6 Multi-Element Scatter Diagram

## ■ State Analysis of Compound Phase

EPMA enables state analysis of microscopic areas, and it is known that state analysis of nickel is possible from the Ni-L  $\beta$  / Ni-L  $\alpha$  ratio and peak wavelength shift by using the Ni-L line. In the Ni-L line spectrum for the Ti<sub>4</sub>Ni<sub>2</sub>O compound phase in Fig. 7, the peak wavelength and ratio differ slightly from those of elemental nickel, indicating that the compound has formed.

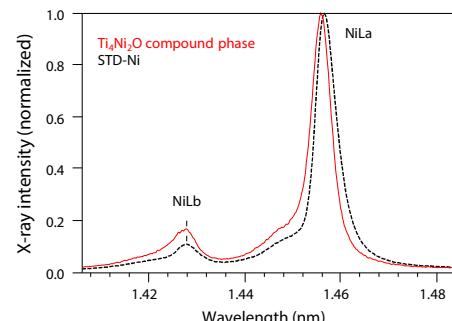


Fig. 7 Ni State Analysis

### <Reference>

The Japan Titanium Society, Metal Materials Utilized at the Site Series: Titanium (2007)

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