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A High quality EBSD pattern from a steel cord prepared with Cross Section Polisher: a newly developed cross-sectioning apparatus

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Minute grains are used in the production of high strength steel cords, such as those used in automobile tires. The size of these grains in the wire-drawn pearlite microstructure is about 10 nm [1-2]. Until quite recently, these microstructures have been analyzed with TEM so that the analysis of the entire cross-section of the steel cords was very difficult and time consuming. It is true that SEM is superior in analyzing in larger areas, but specimen preparation was a serious obstacle for the application to the steel cords of SEM, such as backscattered electron imaging (BEI) and electron backscattered diffraction (EBSD). Because EBSD patterns are formed from the volume element within the depth of 10-50 nm from the surface, preparation of high quality cross-section of specimens is prerequisite for EBSD analysis. The conventional method of mechanical grinding and polishing is usually used as a standard preparation method for EBSD. Unfortunately, however, this method could not produce a high quality cross section of the steel cords for SEM to reveal the microstructure. Recently, we have developed a new cross sectioning apparatus based on milling with an Ar ion beam using a shield plate [3] whose apparatus can produce a very good quality cross section of specimens for high spatial resolution microscopy and microanalysis. This apparatus is called Cross Section Polisher (CP) [4]. This report describes the power of CP for preparing a good quality cross section of a steel cord for the EBSD analysis.

Samples for EBSD analysis were prepared by two methods: the conventional method of mechanical grinding and polishing and the method of ion milling with CP. In the former method, SiC grinding papers with a series of grit sizes (400, 800, 1500) were used, followed by final polishing with colloidal silica with a size of 0.04 μm. The principle of the latter method is schematically shown in Fig.1. No special preliminary preparation of the specimen is necessary for CP. For the present preparation, the accelerating voltage of ion beam was 6kV, the ion beam current was 140 μA and the processing time was 3h. JSM-7000F equipped with a shottky type Field emission gun was used for SEM and EBSD patterns were analyzed with the HKL Technology EBSD system (Oxford instrument).

Fig.2 shows EBSD patterns from a steel cored sample prepared by the two different methods. The EBSD pattern of the sample prepared by the conventional method of mechanical grinding and polishing (Fi.2 (b)) is not sharp enough to draw an EBSD map of recognizable microstructures. The EBSD pattern of the sample prepared by the CP (Fig. 2(a)) is, on the other hand, very high in quality to produce a high spatial resolution EBSD map shown in Fig.3. In this figure, elongated pearlite grains in the longitudinal direction of wire drawing are clearly revealed. Even those with diameters of smaller than 50 nm are clearly observed. This type of analysis with higher spatial resolution in larger areas may serve as a new tool for a quality control of steel cords.

In this report, we showed the preparation of a good quality cross section of a steel cord by CP for SEM, whose section had previously been very difficult or almost impossible to be prepared by other methods. We now believe CP became an established tool for preparing cross sections in good quality for high spatial resolution microscopy and microanalysis of very wide range of specimens.

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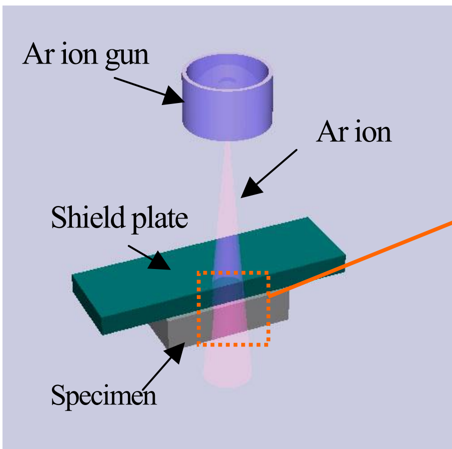
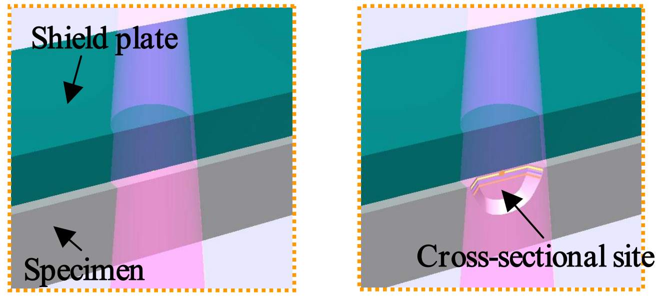
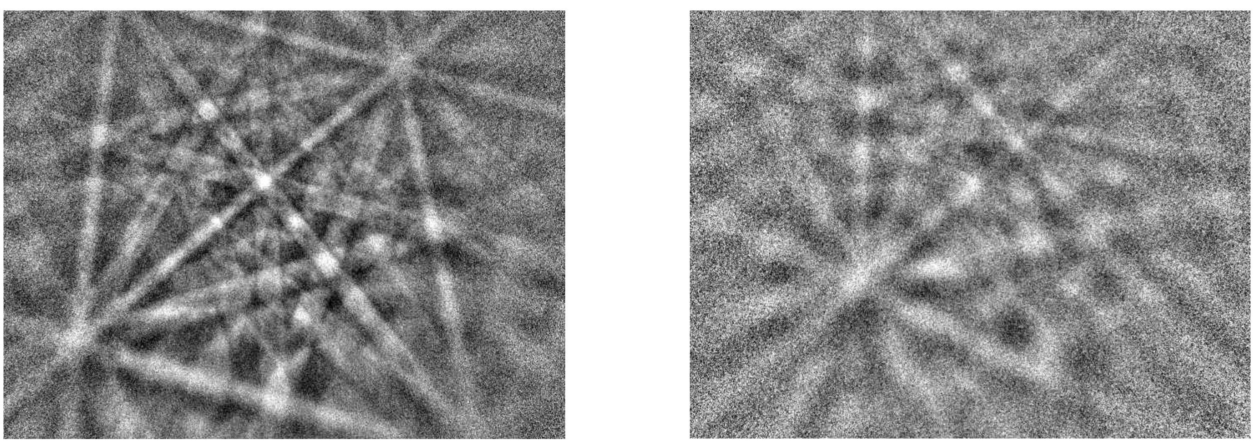




The specimen on the unmasked front side below a shield plate is milled to produce a cross section of the specimen by an Ar ion beam. Because the ion beam is irradiated parallel to the surface of the cross section, milling rate is almost independent of constituents in the specimens and radiation damage due to the ion beam is minimized, to produce a very high quality smooth section with minimum artifacts.

Fig.1 Principle of Cross Section Polisher

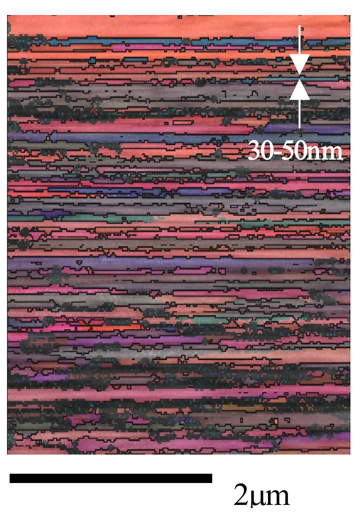


(a) CP (b) Mechanical grinding and polishing

Fig.2 EBSD patterns from a steel cord sample prepared by two different methods

Fig.3 An EBSD map of a steel cord sample prepared with CP

0.02μm step grid 270×203



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