

Trends in Product Developments of Bars and Wire Rods

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Abstract

Outline of trend in development of steel bar and wire rod products is guided. There is a certain tendency towards better function or higher strength of steel products, fewer manufacturing process from customers' needs with increasing importance of global environment preservation. To satisfy these requirements, Nippon Steel has been keeping on supplying new products of high performance. Authors introduce several examples of developed bars and wire rods of special steel, high carbon steel wires and the latest nano-level materials characterization techniques which supports microstructure control of these products.

1. Introduction

In consideration of global environmental issues, steel bars and wire rods are required to enable decreases in the use of environmentally harmful substances, decreases in automobile weight to achieve lower fuel consumption and decreases or elimination of heat treatment in their post-processing into final products. With this as a backdrop, these steel products are strongly required to have better functional properties and higher strength and to allow elimination or simplification of post-processing steps. Nippon Steel Corporation has developed a variety of bar and wire-rod products that meet these widely varied requirements. This paper outlines the trends in new product developments of special-steel bars and wire rods and high-carbon steel wires, and the latest achievements in materials characterization techniques on an atomic scale that support product development activities.

2. Trends in Developments of Bars and Wire Rods of Special Steels

Bars and wire rods of special steel are used extensively for the components of automobile engines, drive lines and chassis, supporting the fundamental performance of automobiles, namely to run, steer and stop as desired. Most of these automobile components are manufactured from steel bars or wire rods through post-processing such as forging or machining into desired shapes and heat treatment to

give desired strength. Thus, these bars and wire rods are required to be capable of ensuring good performance of the machine parts for which they are used, and at the same time, to have good workability at different stages of the post-processing. Japan is by far at the world forefront in terms of special steel technology to realize both high product performance and workability at processing in the manufacture of automobile parts in accordance with economical rationality. For example, an investigation into the world trend in the development of carburizing steel, indispensable for the components of automobile drive lines, over the last 20 years based on technical literature showed that Japan was responsible for an overwhelmingly large share of new products of carburizing steel; 70% developed in Japan versus 14% in Western countries. Especially in the theater of research and development for strength increase of special steels, Japan is virtually the only player.

Thus, it would be fair to say that Japanese special steel suppliers with their technical lead have supported the Japanese automobile industry in its path to the present position in terms of the quality and economical performance of the product as a principal footing. Now Japanese car builders are rapidly expanding their overseas production, and some of them study the use of locally produced special steels. Use of steel materials having inferior quality and economical performance, however, may lead to a decline of the Japanese car industry. To maintain the industry at the present position of technical superiority on the global stage, the steel industry of Japan will have

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to continue supplying high-quality and economically competitive special steel products all over the world. Hence, the importance of our continued efforts for developing new and improved steel products is increasing.

Currently, there are three principal requirements for steel bars and wire rods of special steel for automobile applications: higher strength, elimination or simplification of post-processing, and less use of environmentally harmful substances. Higher steel strength is required because of the need for lower car weight to reduce fuel consumption for conservation of global environment, higher power of automobile engines, smaller number of car parts and wider use of common units, cost reduction and many other reasons. As stated earlier, in the manufacture of car parts, steel bars and wire rods undergo different kinds of secondary working such as forging, drawing and machining as well as heat treatment for annealing, quenching, tempering, etc. Therefore, requirements from the viewpoints of cost reduction, energy saving and environmental conservation strongly urge elimination or simplification of heat treatment and machining (such as near net or chipless shaping).

Control of non-metallic inclusions, precipitates, transformation and metallographic structure is a key metallurgical measure for the property enhancement of special steel bars and wire rods. The control of non-metallic inclusions is one of the main seed techniques for strength increase of spring steels, service-life extension of bearing steels, elimination of lead from free-cutting steels, etc.; control of the structure and shape of oxides and sulfides is effective in realizing desired properties of these steels, and further studies in this direction are carried on.

Control of precipitates is utilized for two purposes: control of austenite grain size and precipitation hardening (see Fig. 1). With a wide variety of car parts such as suspension arms, tie rods, springs, gears, and bolts made of microalloyed steel for hot forging and gear steel for carburizing, refinement of austenite grains is effective in increasing their toughness and fatigue strength as well as decreasing strain and improving resistance to delayed fracture. Fig. 2 shows anti-coarsening extra-fine steel for carburizing as an example of new products utilizing control of crystal grain size. The essential for grain size control is to have fine precipitates that serve as pinning particles dispersed in the steel at the final heat treatment stage, and in this sense, precipitate control in all the processes of parts manufacture is of great importance¹⁾. Note that precipitates are used also as the intragranular ferrite nuclei to refine the structure; such an example is microalloyed steel for hot forging (see Fig. 3)²⁾.

Precipitation hardening, on the other hand, is used in two different ways: one is precipitation at interfaces between γ and α phases

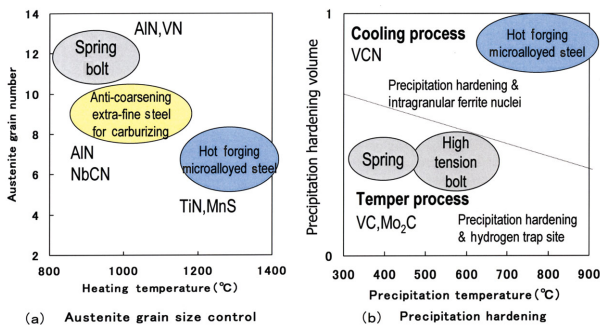


Fig. 1 Effective use of precipitates for improvement of special steel bar and wire rod products

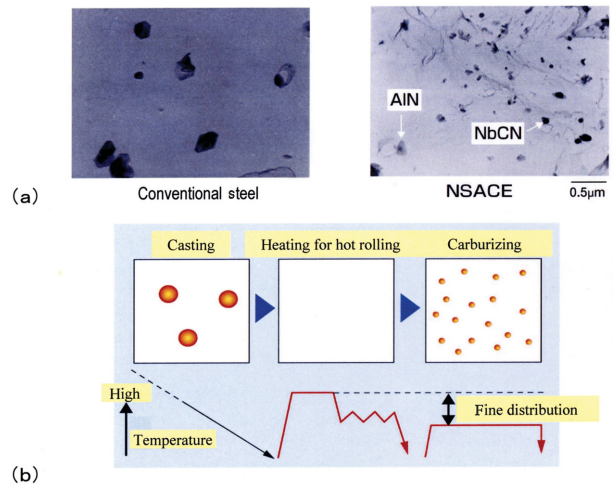


Fig. 2 Anti-coarsening extra-fine steel for carburizing, utilizing pinning effect of precipitates for suppressing grain coarsening

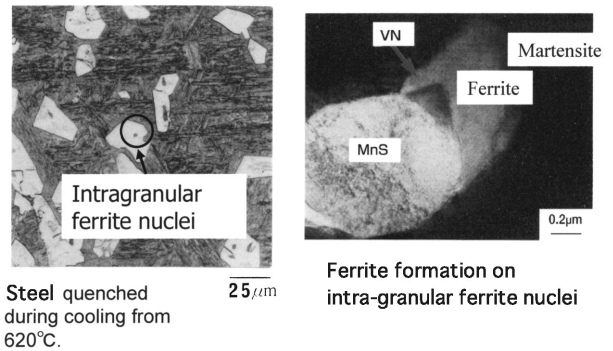


Fig. 3 Structure control utilizing formation of intragranular ferrite on composite precipitates serving as transformation nuclei

during the cooling stage after hot forging as in the case of microalloyed steel for hot forging, and the other is precipitation during tempering as in the case of bolt and spring steels. Precipitates have been used for various purposes such as increasing strength and endurance ratio, suppressing temper softening, and trapping hydrogen to improve resistance to delayed fracture³⁾. The use of precipitates is expected to expand for many other purposes.

The control of transformation and metallographic structure is a key metallurgical means for objects such as increased toughness of microalloyed steel for hot forging, softening of steel for cold forging and elimination or simplification of annealing⁴⁾. What is attracting attention recently as application of the transformation and structure control is the possibility of developing a new steel product for machine parts incorporating in its material some functions of post-processing, such as crystal grain refinement and material property control by controlled forging. Chipless shaping is also strongly required, and the development of a steel product for near net shaping to meet the need is another important challenge for the steel industry.

The need for higher strength of car components to reduce fuel consumption is very strong, and grain boundary strengthening is an important means for this purpose. The structure of most car components made of special steel bars and wire rods is tempered martensite, and the grain boundary strength being discussed in this filed of industry is that of pre-austenite grains. The mode of fracture at the impact failure of springs, delayed fracture of bolts, fatigue or static

fracture of carburized and induction-hardened steels used for gears, shafts, etc. is usually intergranular fracture. Therefore, increasing grain boundary strength to prevent the intergranular fracture leads directly to higher strength of these parts.

Fig. 4 shows an example of prevention of delayed fracture of high-strength bolts, wherein intergranular fracture is prevented by measures such as increase in grain boundary strength⁵⁾. Boron addition is attracting attention as a principal technique for increasing grain boundary strength of surface-hardened parts. This effect results from the decrease in the segregation of P at pre-austenite grain boundaries, which is due to the site competition between B and P, as shown with the result of Auger electron spectroscopy (AES) in Fig. 5⁶⁾. On the other hand, Fig. 6 shows the decrease in the segregation of P at grain boundaries resulting from refinement of γ grains. It is clear from the graph that crystal grain refinement is the principal measure for grain boundary strengthening, or increase in steel strength, and further refinement of crystal grains will be one of the main approaches in the research and development to improve the strength of machine parts. In this relation, the relationships of grain boundary strength with factors such as the shape of grain boundary cementite and tempering temperature are newly attracting attention, and further studies into grain boundary strengthening from different approaches are awaited.

On the other hand, recent changes in automobile engines and drive lines require car components having properties not strongly required in the past. These newly required properties include higher function-

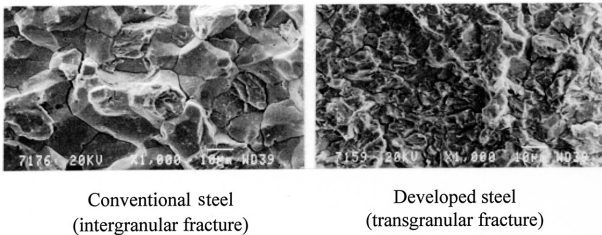


Fig. 4 Delayed fracture surface of high tensile strength bolt

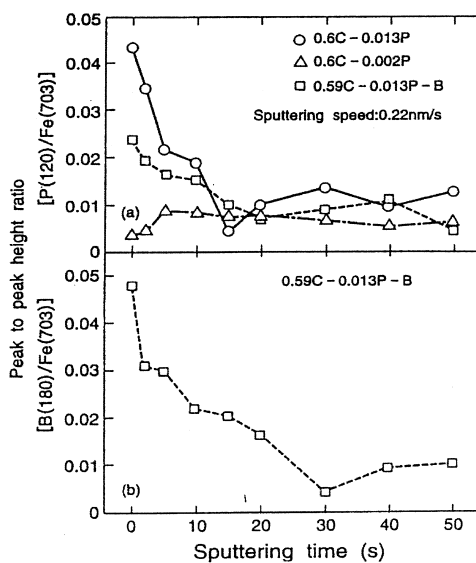


Fig. 5 Grain boundary segregation behavior of P and B at the pre-austenite grain boundary of middle-carbon martensitic steel

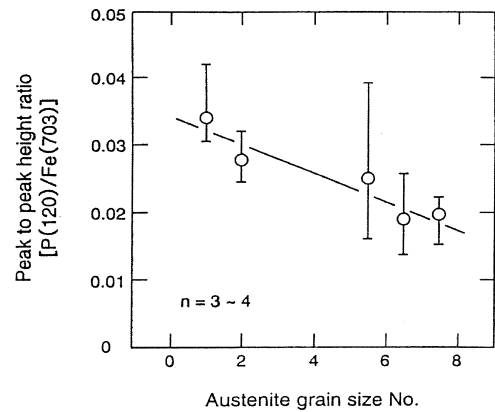


Fig. 6 Relationship between γ - grain size number and the amount of segregation of P at grain boundaries

ality of common rail parts due to wider use of diesel engines, lower strain and higher pitting resistance of planetary gears for increased the number of gears of automatic transmissions, good high-temperature carburizing properties of material for the pulleys of continuously variable transmissions (CVTs), the use of which is now rapidly expanding. Surface-hardened steels are commonly used for these applications, and the typical methods of surface hardening are carburizing, nitriding and induction hardening. Each of these methods has its advantages and shortcomings in terms of hardness distribution, strength, strain and productivity. Besides selecting the most suitable among these methods, combination of two or more treatment methods and application of surface reforming treatment such as double shot peening are expected to be effective for improving the strength of these parts. What is important with respect to strain reducing of gears is clarification of factors that induce strain in all the parts manufacturing processes from steel material through tooth design and machining to heat treatment, especially clarification of the influences of the medium and conditions of cooling in heat treatment and improvement in the accuracy of strain simulation techniques.

3. Trend in Development of Wire Rods of High-Steel

Wire rods of high-carbon steel shipped from steel mills is worked into high-strength wires through secondary working steps such as wire drawing and patenting treatment at a secondary working industry. Wire rod products are classified roughly into two categories with the difference in production process, which is determined by strengths and diameters of the final wires: one is thick wire rods 10 to 14 mm in diameter which are worked into wires 3 to 7 mm in diameter for applications such as PC wires, galvanized wires for bridge cables, and ropes; and the other is thin wire rods 5.5 mm in diameter which is drawn into fine wires usually 0.15 to 0.35 mm in diameter, which process require patenting treatment between steps of drawing, for applications such as tire cords and saw wires.

3.1 Thick wire rods

In consideration of global environmental issue, many wire drawers of thick wire rods closed their lead patenting facilities and began to use direct-in-line-patenting (DLP) wire rods treated patenting equivalent to off line lead patenting, immediately after hot rolling at wire rods mills. The use of DLP wire rods not only allows elimination of the environmental measures against the use of lead but also makes it possible for wire drawers to omit a processing step or two to reduce their costs. Since Nippon Steel's DLP wire rods manufac-

tured through a direct patenting process using a molten salt bath^{7,8)} realizes the level of strength and ductility conventionally obtainable only through lead patenting without altering chemical compositions, most of the thick wire rod products in Kimitsu Works can be replaced with DLP wire rods resulting from studying for about 20 years after the introduction of the DLP equipment in 1985. As the use of DLP wire rods expanded, the importance of integrated quality control including the secondary processing stages increased. And it has become necessary for product development activities to conduct consistent research and development from steel material design to final wire products in an integrated manner.

The 7 mm dia.-1800MPa steel wire for bridge use and 2300-MPa PC wire, which are presented in other articles in this Special Issue, were developed as a result of joint studies with Tokyo Rope Manufacturing Co., Lit., Suzuki Metal Industry Co., Ltd. and Sumitomo Steel Wire Co., Lit. to meet extremely demanding quality requirement. While higher and higher steel strength is in demand, research and development activities will be focused on steel material design assuming the DLP process and on the engineering of DLP equipment suitable for the material design.

Another essential issue in development of higher-strength wire is a problem about resistance to delayed fracture. Although generally pearlite steel after drawing shows higher resistant to delayed fracture, the higher strength of such steel wire may be raised to a cautious level⁹⁾ where delayed fracture occurs easily. So basic metallurgical research should focus more on delayed fracture properties of the wire.

3.2 Thin wire rods

The global market of thin wire rods for application such as steel tire cord is expanding as the production of automobile in China increases and the market is becoming highly competitive. The important feature demanded in the thin wire rods has been high productivity in secondary working and high strength after final drawing. And the feature will continue to be important. The high productivity means minimizing wire breakage during drawing and bunching or ideal no breakage, higher direct drawability, and good drawability under severe drawing condition such as high drawing speed. Nippon Steel has made efforts to meet these increasingly sophisticated requirements of customers by applying stringent standards to quality control items such as non-metallic inclusions, segregation, decarburizing, mechanical properties and microstructure. In addition, beside developing new steel products, we have developed various secondary working techniques for users of our products to bring out the most of the performance of the wire rods^{10,11)}.

An example of high-strength wire rod of Nippon Steel is wire rods of hypereutectoid steel¹²⁾, the strength of wire drawn from which is shown in Fig. 7. We have also developed techniques to inhibit delamination^{11,13)} as shown in Fig. 8. Although it has been 12 years since the hypereutectoid steel wire rods were launched to the market, the share of 90C-class wire rods in whole market of steel tire cords is still small. While there has been a remarkable technical advancements regarding the secondary working for 80C materials. And now, the technical conditions could be sufficiently ripe for expanded use of 90C wire rods.

Fig. 9 shows photographs of microstructures obtained through wire drawing of pearlite steel taken with a transmission electron microscope (TEM). As an amount of working increases, cementite, becomes unclear and the lamellar structure also becomes unclear.

Recently we think that material science of heavily worked pearlite has been advanced notably. After those were reported that ce-

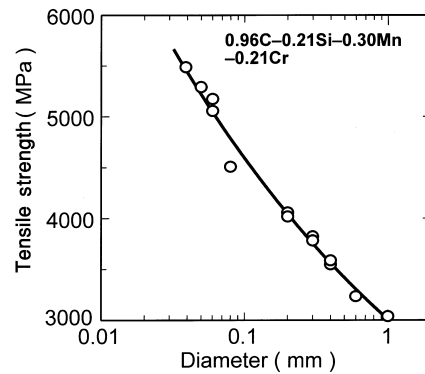


Fig. 7 Relationship between diameter and tensile strength of hypereutectoid steel wires

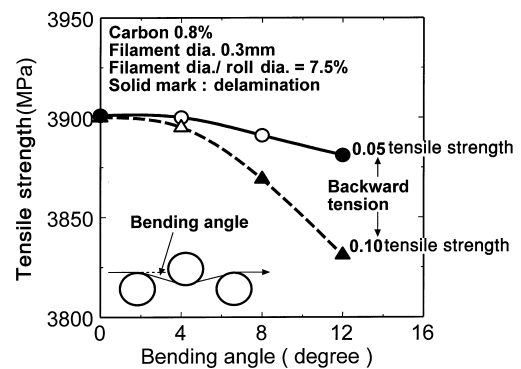


Fig. 8 Effect of roll bending on tensile strength and delamination of fine filament

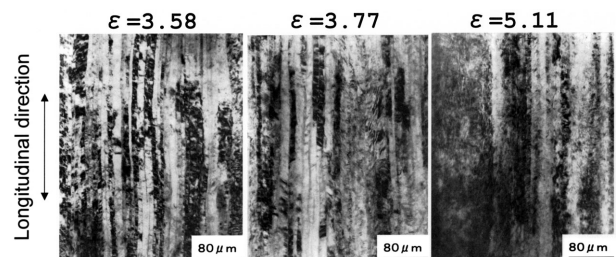


Fig. 9 TEM micrographs of 0.96% carbon steel wires (ε : drawing strain)

mentite dissolve in heavily worked pearlite¹⁴⁾ and that supersaturated carbon atoms exist in ferrite¹⁵⁾, several findings in order to make clear strengthening mechanism of heavily worked pearlite has been reported by researchers belonging to university or other research institute. Those are physical or mechanical properties of bulk cementite¹⁶⁾, stress distribution between cementite and ferrite of pearlite during tensile test^{17,18)} and so on.

Tire makers are demanding strongly a higher strength steel tire cord for the purpose of reducing car weight and tire weight. Therefore we will not only study process condition producing a steel tire cord of strength attained in only lab scale but also conduct basic research¹⁹⁾ how to attain higher strength than ever.

4. Analysis Techniques Supporting Development of High-grade Bars and Wire Rods

As the market of steel bars and wire rods expanded globally, requirements of their users became widely varied, and as a result, mutually incompatible properties have come to be required for product development. For example, simple pursuit of higher strength for weight reduction does not meet the requirement for better ductility and formability, nor does it satisfy the need for corrosion resistance in different environments or that for combination with wider variety of materials. In trying to satisfy such mutually incompatible requirements, an important approach is to clarify the intrinsic physical properties of the material in question and describe widely varied phenomena applying physical models. Since materials science pursues truth based on the findings obtainable within the capability of analysis techniques available that can describe the phenomena, increasingly advanced analysis techniques are being looked for and worked out in parallel to the development of new products. Some examples of the recent evolution of analysis techniques that support the development of steel bars and wire rods are explained below.

4.1 Technical advancement in nano-structure characterization based on transmission electron microscopy

Metallographically speaking, steel wires for bridges and steel tire cords consist of pearlite, the same structure of high-strength and highly ductile piano wires, and these products are given their respective characteristic strengths by controlling the thicknesses of ferrite and cementite layers (lamella thickness) that form the lamella structure of pearlite. The lamella thickness is about 50 nm in the case of a steel wire for bridge use 5 mm in diameter, and about 10 nm in the case of a steel tire cord approximately 0.2 mm in diameter; analysis of such a fine lamella structure is possible only by using a transmission electron microscope (TEM) equipped with a field-emitting electron gun capable of focusing an electron beam to a diameter of several nanometers²⁰⁾.

On the other hand, it is important to understand how a pearlite structure deforms through wire drawing for studying the ductility of a steel tire cord in downstream process steps. In a drawing process, the lamella structure is stretched but maintained when viewed in a longitudinal section, and it is of more interest to observe the change of the structure in a transverse section. In the case of a fine wire 0.2 mm in diameter, for instance, however, it is not easy to prepare a thin specimen for TEM observation to examine its microstructure at a section in right angles to the direction of drawing. A method was proposed whereby several thin wires were bundled and embedded in resin, and a thin specimen that showed the sections of the bundled wires was fabricated, but by this method, the wires often fell out during the process to make the specimen thin enough for TEM observation. All these problems were fundamentally solved by the micro-sampling method the equipment of which could be installed inside a device for focused ion beam (FIB) fabrication²¹⁾.

Fig. 10 (a) is a scanning ion microscope (SIM) image of a thin wire taken inside a FIB fabrication device. When observing an area tens of micrometers across even of a thin wire 0.2 mm in diameter, the field of vision is substantially flat, and it is possible to select a section in right angles to the drawing direction at a desired position using a manipulator provided in a FIB device like in the case of a flat specimen, as seen with Fig. 10 (b). A FIB device is a facility similar to a scanning electron microscope, but it uses a Ga ion source instead of an electron gun, focuses a Ga ion beam on a desired portion of a specimen using an electrostatic lens, machines the portion according to the principle of sputtering, and thus forms the specimen

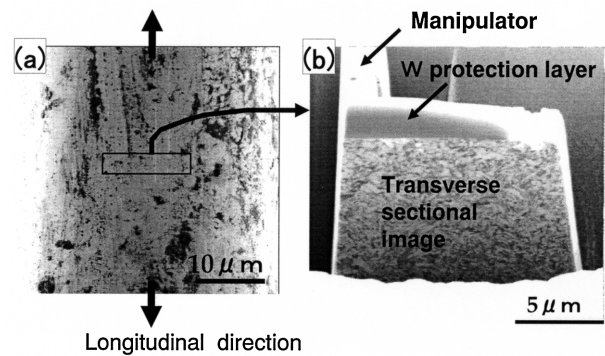


Fig. 10 TEM sample preparation procedure using FIB micro-sampling, especially from the transverse sectional view of steel cord 0.2 mm in diameter
(a) Selection of objective area by scanning ion microscope observation, (b) Selection of a structure in a transverse section using micro-sampling manipulator

portion into a desired shape. Note that a thin protective layer of W is formed on the surface of a specimen inside the FIB device to protect it against damage by the Ga ion beam. Since a SIM image exhibits channeling contrast related to crystal orientation, it is possible to study a pearlite structure deformed through drawing.

More specifically, such a pearlite structure is examined using TEM images, as in Fig. 11, taken after fabricating a specimen into a thin plate 0.2 μm or less in thickness using a Ga ion beam. The photomicrograph shows that, judging from the transverse sectional shape of the dark portions, which are mostly cementite layers, the structure has been twisted considerably by the wire drawing. In addition, the portion to the right from the center of the photograph seems to have undergone a shear deformation. Thus, it is possible to analyze if the deformation by wire drawing occurred homogeneously across the whole diameter. When studying the effects of heat generation during drawing work, it is important to compare the structures at the center and peripheral portions; this has been made practicable owing to the advance in electron microscopy employing these new micro-sampling techniques.

On the other hand, different from electrolytic polishing, selective etching of different metal species hardly occurs by the FIB technique, and consequently, it became possible to observe details of the interface structure between base steel and a brass coating layer with a TEM. Fig. 12 is a TEM micrograph of a section in the drawing direction of such an interface; the portion exhibiting a lamella structure is the base steel of pearlite and that in an oblique twin structure

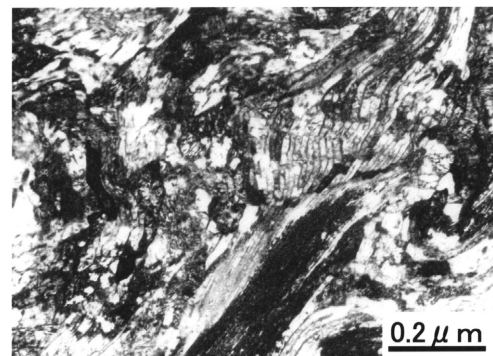


Fig. 11 Transverse sectional TEM micrograph of fine steel cord

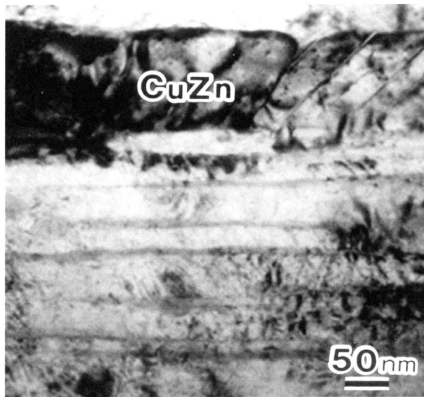


Fig. 12 TEM micrograph of brass plating interface of fine steel cord

is the brass coating layer. The adhesion between the two is so good that the interface is not clearly distinguishable. Another advantage of the FIB machining technique is that a composite material can be machined evenly with no regard to the difference between metal and resin. It is also possible, therefore, to examine delicate structural change of a steel tire cord after it is embedded in rubber and vulcanized.

As explained above, FIB machining is not significantly affected by the difference between material kinds, and FIB micro sampling is capable of selecting a position desirable for observation with a pin-point accuracy and forming a thin specimen exactly at the selected position. The development and advance of these techniques have made it possible to analyze fine structural details not only of steel tire cords but also of portions in complicated shapes of nitrided car parts or high-strength bolts made of steel bars using a TEM and atom probe micro-analyzer.

4.2 Technical advance in atomic-scale analysis using atom probe technique

The combination of FIB machining and TEM has made it possible to prepare a specimen for TEM observation at a desired position comparatively easily, and as a result of accumulated microscopic findings obtained from portions hitherto impossible to observe, need for more advanced analysis techniques arose. In other words, the need for higher-strength steels urges clarification of metallographic phenomena in more detail, for which atomic-scale material analysis is sometimes required. The 3D atom probe method is a technique to directly observe the atomic distribution of a steel material. The recent development of an energy compensator has improved the mass resolution capacity of the method²²⁾. The principle of the atom probe is as follows: a high-voltage DC and pulsating current is applied on a needle-shaped specimen, to make the atoms at the surface of the specimen ionize and evaporate layer by layer, and the masses and original positions of the ionized atoms are detected. We have established a technique to draw a three-dimensional atomic mapping of a steel specimen in a real space through introduction of the 3D atom probe method and development of an analysis technique that could visualize the atomic distribution information.

Nitriding is a kind of heat treatment to harden the surface layer of a steel material. An analysis on an atomic level disclosed that new type of composite precipitates of Cu and CrN formed when steel containing Cr and Cu for automobile use underwent nitro-carburizing treatment²³⁾. Here, with respect to surface hardening effect, whereas the base steel hardness before the treatment was somewhere around 100 HV_{0.1}, that of its surface layer increased through the treat-

ment to roughly 700 HV_{0.1}. TEM observation focusing on a region at a depth of 160 μm from the surface, near the position of peak hardness, revealed contrast lines 6 to 10 nm in length along {001} planes as shown in Fig. 13.

Elemental analysis of some of the precipitates using an electron beam focused to a diameter of 1 nm identified them as chromium nitride. Whereas electron microscope observation had revealed many tabular nitride precipitates, which were then found to harden the steel, atom probe examination of the spatial distribution of atoms using a needle-shaped specimen made it clear that the nitride often formed composite tabular precipitates together with Cu.

Fig. 14 is an elemental atom mapping obtained by the 3D atom probe method. What is seen here along (010) and (100) planes is CrN precipitates. The 3D atom probe technique is characterized by the ability to detect atoms virtually without elemental selectivity. It is, therefore, possible to estimate the number of solute atoms and the density of precipitates in the whole bulk of a specimen by measuring the numbers of atoms and precipitates directly from an atomic distribution image obtained through the 3D atom probe method. The 3D atom probe measurement clarified that the density of CrN was on the order of as high as 10¹⁸ cm⁻³ and that of the Cu precipitates was on the order of 10¹⁷ cm⁻³. Especially through sequential atom probe analysis in the depth direction, CrN was found to precipitate selectively, around precipitates of Cu that had deposited during nitriding treatment. Because the density distribution of these precipitates directly affects the degree of steel surface hardening, observing the processes of the formation and growth of the precipitates during nitriding treatment is of great importance²⁴⁾.

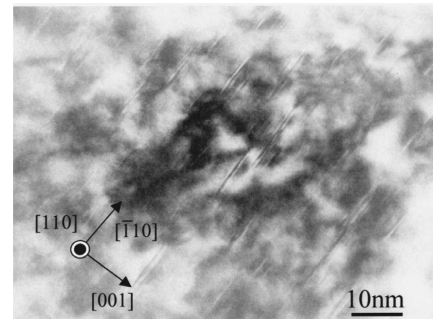


Fig. 13 TEM micrograph of nitriding steel at 160 μm depth from surface after nitro-carburizing treatment

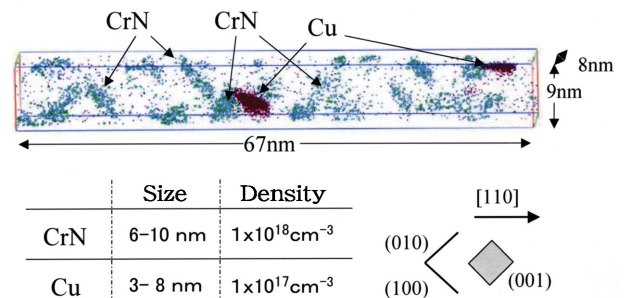


Fig. 14 3-dimensional elemental map in region at 160 μm depth from surface in copper-added nitriding steel. Measurement direction is [110]. Size and densities of each precipitate calculated by 3-dimensional measurement are listed

4.3 Future prospects

As the quality requirements for steel bars and wire rods become more and more sophisticated, need is increasing for fundamental researches to solve difficult problems such as compatibility of high strength with good ductility, and prevention of brittle fracture due to hydrogen, and as a result, application of the most advanced analysis techniques is strongly required. It is necessary for production of industrial materials to clarify phenomena from the macroscopic structural changes to those on an atomic scale in an integrated manner. While nanometer-scale measurement technology showed significant advance, practical mesoscopic technique to prepare a specimen at a desired position was looked for. Thanks to the development of such technology typically such as FIB, the understanding of material structure has become more integrated. Now, the three-dimensional atom mapping by the atom probe technique is intensively applied to the researches of commercial steel materials. Analysis technology and development of high-performance steel products will continue to advance together, one spurred by the other.

5. Closing

This paper has presented Nippon Steel's product development activities in the field of bars and wire rods in response to ever increasing customer requirements for enhanced product performance and higher strength. While the customer requirements for product quality become sophisticated year by year, it is increasingly important to make the best of material characteristics in the processes from the production of steel products through their post-processing to the manufacture of final products. Nippon Steel will continue developing new products exercising its advanced technical capability and in close cooperation with customers.

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