Metallographic structure control technology

Metallographic Structure Control Technology Contributing to Development of New Metallic Materials to Meet Social Demands

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Abstract

Kobe Steel, as a comprehensive manufacturer of metal materials, has been advancing the refinement of metallographic structure control technology as a fundamental technology to enhance the performance of various metal materials, including steel, aluminum alloys, copper alloys, titanium alloys, and welding materials, in order to meet the diverse demands of society. This paper presents examples of the company's initiatives: the development of ultra-high-strength steel and aluminum alloys for automotive applications as part of its efforts towards weight- reduction solutions that contribute to a sustainable society, and the development of thick steel plates and welding materials, working toward the creation of safe and secure communities. These examples serve to showcase the application of the metallographic structure control technology. Furthermore, the paper discusses the future direction of this technology as an effective response to anticipated significant societal changes.

Introduction

All products have one aspect in common: They are made up of individual materials. Multiple perspectives determine which materials are used, including the product's application, performance level, function, and cost. Metal materials in particular feature a wide variety of performance attributes such as strength, ductility, toughness, and electrical and thermal conductivity. They also exhibit favorable performance characteristics at a relatively low cost even when produced in large volumes. Therefore, although periodic surges in material advancements affect trends in the application of materials, metal materials always carry an important role that supports societal needs. Metal materials continue to be used not only because of their superior performance, but also because they have undergone continuous improvement to meet the increasing demands of structural change within society.

Kobe Steel produces a comprehensive variety of metal materials for use in end products including steel (wire rod and bar, steel plate and sheet, cast and forged steel products, iron powder), aluminum alloys (aluminum sheet, aluminum cast and forged products, aluminum extrusions), copper alloys, titanium alloys, and welding consumables. Industries we supply include automotive, railroad vehicle, shipbuilding, aircraft, transportation equipment, construction, architecture, industrial machinery, electrical machinery, and electronic components, devices, and circuits. The metal materials used in these products must meet particular performance requirements. Achieving both high strength and favorable deformation characteristics such as formability in metal materials fosters the lightweight design of transportation equipment such as vehicles. Such an achievement reduces CO_2 emissions by improving fuel efficiency, thus contributing to a sustainable society. Moreover, improving the reliability of structural materials, such as through enhanced toughness and fatigue properties, increases the reliability of the end product. This in turn contributes to a safer and more secure society.

The performance of a metal material is governed by both its composition and its microstructure, the crystalline distribution on the order of microns to nanometers. Hence, metallographic structure control technology for fine-tuning the performance of various metal materials is a pillar of the competitiveness of Kobe Steel's metal material products.

Metallographic structure control technology steers the microstructure of a metal in support of the requisite target properties. Composition, processing methods, and heat treatment are some of the parameters manipulated. Automobile frame parts are one application in which metallographic structure control technology is critical. These formed parts have complex shapes that deform to absorb energy during a collision, necessitating high strength and ductility at the same time. To meet these requirements, microstructure control technology is used to combine high-strength, low-ductility microstructures with low-strength, high-ductility microstructures, or to control the orientation of metal crystals so they are easily deformed. Structures that must not fracture during use, such as buildings and ships, require structural reliability in the form of favorable toughness and fatigue properties. As such, the microstructures of these structures must be controlled to suppress fracture, such as through uniformity of the metallographic structure and refinement of the crystalline grain.

We have developed control technology to achieve target microstructures amidst challenging parameters. Examples include high-temperature strength and creep resistance for materials in high-temperature environments, electromagnetic properties and thermal conductivity in functional systems, and high strength alongside the contradictory properties of hydrogen embrittlement resistance, drawability, and workability.

This report introduces some of our metallographic structure control technology that support weight reduction solutions for a safe, secure, and sustainable society.

1. Metallographic structure control technology for weight reduction solutions in support of a sustainable society

Achieving a sustainable society requires the reduction of CO_2 emissions to mitigate global warming. One of the most important objectives is to reduce CO_2 emissions from transit, which accounts for nearly 20% of Japan's CO_2 emissions, and particularly from vehicles. Reducing CO_2 emissions caused by vehicle operation requires improvements in fuel efficiency by reducing vehicle weight. As a materials manufacturer, Kobe Steel has been developing weight reduction technologies in support of this goal. As a central part of this effort, we have been enhancing our metallographic structure control technology to create lightweight materials.

One such example is the development of ultra-high-tensile strength steel sheets for vehicles. Frame parts must exhibit high strength to ensure crashworthiness alongside reduced vehicle weight. To this end, we have been developing technologies to design steel sheets that feature high strength and formability. The 1980s saw research into dual-phase steels (DP steels), which combine soft ferrite and hard martensite, to achieve both strength and ductility.[1), 2)] We developed microstructure control technology for DP steels to ensure strength alongside elongation and stretch-flangeability, ductility factors that are considered to be incompatible with strength. Specifically, the balance between elongation and stretch-flangeability is improved by controlling the ferrite and martensite fractions, carbon concentration in martensite, and carbide size in martensite.[3), 4)]

TRIP steels, which exploit the work-induced transformation of retained austenite (hereinafter, retained γ), were considered as candidates to meet the need for greater ductility. However, TRIP steels have good elongation but relatively poor stretch-flangeability. This could be caused by large areas of retained γ. We conducted joint research with Shinshu University to develop microstructure control technology for fine dispersion of the retained γ. Two microstructure concepts were developed as a result: TAM steel (TRIP-aided annealed martensite steel)[5), 6)], in which the pre-annealed structure is martensite with retained γ finely dispersed between martensite laths, and TBF steel (TRIP-aided bainitic ferrite steel)[7)], in which the matrix phase is bainitic ferrite with retained γ finely dispersed between bainitic ferrite. Applying this concept to various strength grades, we have developed materials such as 980 MPa-grade steel (**Fig. 1**),[8)] 1180 MPa-grade steel,[9)] and 1470 MPa-grade steel,[10)] which have an excellent balance between elongation and stretch-flangeability.

Research has also delved into ultra-high-tensile strength steel plates with a relatively low alloy content and an excellent balance between strength and ductility. These ultra-high-tensile strength steels, intended for weight reduction, are known as Generation 3 or 3rd Generation steels.[11)] A project was also initiated in Japan to achieve a tensile strength of 1.5 GPa and an elongation of 20%. For this project, Kobe Steel developed a particular microstructure control technology for medium- to

(a)

(b)

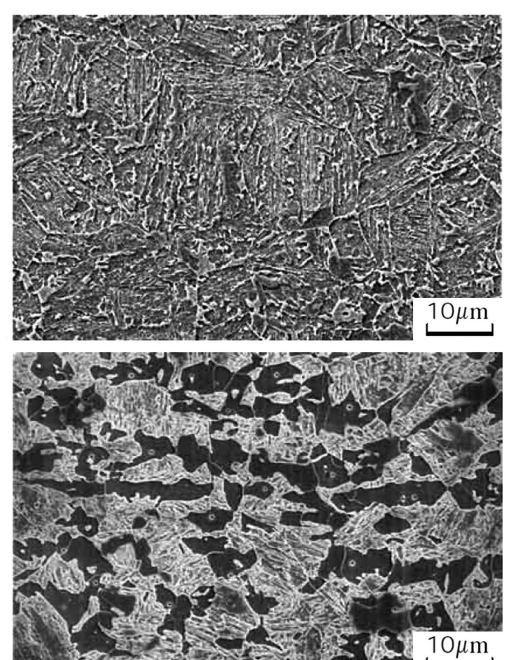

Fig. 1 SEM images of microstructure of (a) 980 MPa-grade steel developed (TBF steel), (b) conventional 980 MPa-grade steel (DP steel)

high-carbon steel. The technology increases and controls the stability of retained γ[12] and uses the Q&P process to refine the microstructure, thereby achieving the target strength/elongation ratio and ensuring stretch-flangeability.[13] Although various challenges must be overcome before the practical application of this microstructure control technology, it will lead to steel sheet innovations that reduce the weight of end products.

Aluminum alloys are another promising material for reducing the weight of vehicle bodies. Aluminum alloys can exhibit the rigidity of steel at half the weight, which is why they are seeing increased use in panels that demand high rigidity, such as hoods, trunk lids, back doors, and roofs. Panel materials must have high strength, formability, surface quality after forming, and hemming ability (the ability to be folded and then pressed).

Following is an explanation of microstructure control technologies for ensuring strength, particularly after paint-baking, and surface quality after press forming.

Panel materials for the vehicle exterior, in particular, must be able to be formed into highly precise complex shapes, since their dimensionality is critical to both function and design. Therefore, these materials must have low strength during forming, yet the final product must have high strength for dent resistance. Vehicle manufacturing includes a process called paint-baking, in which the product is heated to 170℃ to 180℃ for several dozen minutes after press forming. The bake hardening (BH) index correlates to the increase in hardness resulting from this heating process. Even if a material's strength is low at the time of forming, the product can exhibit high strength if the BH index is high. Hence, research into increasing the BH index is actively being pursued.

Heat treatment via paint-baking increases strength because of the formation of nanoscale structures called precipitates and clusters. Pre-aging or a combination of pre-aging and pre-straining improves the BH index[14), 15), 16)], and the BH index can be maximized by optimizing each of these processes. However, the clusters that govern the BH index are fine microstructure, only a few nanometers, that comprise solute elements (Mg and Si in 6000-series aluminum alloys) and vacancies. They cannot be observed clearly via transmission electron microscopy (TEM), a common method for observing microscopic structures. This is why we used three-dimensional atom probe tomography to observe atomic arrangement in three dimensions and thus analyze cluster formation behavior during heat treatment[17), 18)], as shown in **Fig. 2**. The results show that pre-aging increases the number density of clusters with a Mg/Si ratio of more than 0.4 compared with natural aging, and that the transition to the β phase during aging improves the BH index, which enhances strength.

One surface quality defect that can occur after press forming 6000-series Al-Mg-Si alloy sheets is the formation of a pattern called ridging (or roping). Ridging must be inhibited to use these sheets as the exterior vehicle panels from the perspective of appearance.

The anisotropic nature of metal deformation is one of the causes of ridging. Metal deformation occurs when areas of altered crystalline connections called dislocations form and move. The direction in which dislocations can move is more limited in aluminum than in steel. Materials are easily deformed when stress is applied in the direction in which dislocations can move, and less easily deformed when stress is applied in other directions. This relationship between crystalline orientation and the direction of deformation governs deformability. Ridging is influenced by a texture called a Cube grain, which is effective in improving overhang.

Texture develops when a crystalline orientation that is favorable for deformation forms during hot rolling or cold rolling. Recrystallization then occurs during rolling or subsequent heat treatment, in which crystalline grains with a specific orientation free of work strain predominantly form and grow. We exploited these principles to develop a texture

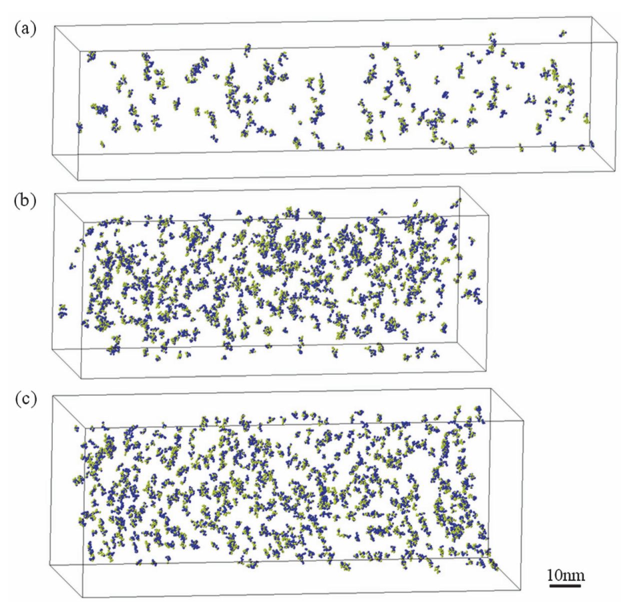

Fig. 2 3D elemental distribution of Mg (green) and Si (blue) atoms of Mg-Si clusters in specimens natural-aged for (a) 10.8 ks, (b) 360 ks, (c) 2.8×10^4 ks, observed by 3D atom probe control technique for the hot rolling process and the subsequent cold rolling[19] and solution heat treatment[20] processes of aluminum manufacturing. We also developed an analysis technique using crystal plasticity theory, which accounts for crystallographic anisotropy, to show that the texture state in which ridging develops is caused by the heterogeneous distribution of Goss and Cube grains (**Fig. 3**).[21] We have developed a microstructure control technique based on this information to develop aluminum panels with good formability and a low propensity for ridging.

We have also contributed to weight reduction in vehicles and have developed various microstructure control technologies that achieve high strength alongside various conflicting properties. Examples include the following: crystalline grain refinement and precipitate control for hydrogen embrittlement resistance and sag resistance of spring steel;[22), 23] precipitate and cluster control to suppress serration in 5000-series aluminum sheets;[24), 25] precipitate control via two-step aging to shorten the pre-aging time of aluminum forgings;[26] recrystallization texture control to improve the impact resistance of aluminum extrusions;[27] and exploitation of dislocation drag resistance through the addition of Ni and P[28] as well as control of the precipitate dispersion state[29] to achieve strength, electrical conductivity, and stress relaxation resistance in copper sheet for automotive electrical connectors.

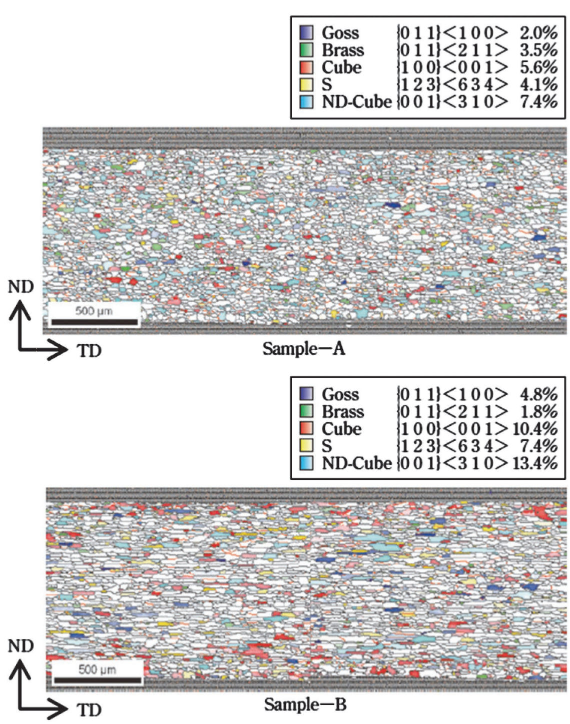

Fig. 3 SEM-EBSD maps of aluminum sheets (a) without ridging and (b) with ridging

2. Metallographic structure control technology that ensuring safety and security in community development

Welding consumables join the steel plates in large structures such as buildings and bridges. The demand for high-strength materials has grown in recent years because of increasing building heights, particularly in urban areas, and the rationalization of steel structures. Furthermore, welding productivity must be increased to reduce construction costs. This necessitates an increase in the thermal energy (heat input) applied during welding and therefore the permissibility of high-heat-input welding, in which a given welding operation uses a large amount of welding consumables.

Ensuring toughness is a challenge in increasing the strength of steel plates and supporting high-heat-input welding. Toughness, a measure of resistance to fracture, is necessary to preclude the brittle fracture of a structure. The SS Schenectady, a United States warship, serves as a well-known case study in catastrophic brittle fracture.[30] Because such failures in large structures have a significant impact on society, it is essential to ensure the toughness of steel materials and welds.

Crystalline grain size is the primary microstructural parameter governing the toughness of steel. This is the order at which brittle fracture propagates; the finer the grain size, the greater the toughness. Refining and reducing the hard second-phase microstructure in the steel, which is the origin of fracture, also improves toughness.

Also of note is that austenite, a high-temperature phase, forms in the heat-affected zone (HAZ) in the weld joint of steel plate. Austenite crystalline grains coarsen as heat increases in the HAZ during welding. During subsequent cooling, coarsening occurs through the formation of structures such as ferrite and bainite at the austenite grain boundaries, reducing toughness.

Therefore, a method of safeguarding the toughness of the HAZ is to suppress the coarsening of austenite crystalline grains at high temperatures during welding. This can be accomplished using the pinning effect of dispersed particles. Another method is to control transformation behavior during cooling to form fine ferrite and bainite microstructures. Kobe Steel has combined the two methods to develop a high-strength steel with excellent high-heat-input HAZ toughness. This was accomplished by using TiN, which is stable at high temperatures, as pinning particles and by developing microstructure control technology that uses low-carbon bainite with a fine microstructure as shown in **Fig. 4**.[31), 32)] In addition, as a method of refining TiN and increasing the number of TiN particles to enhance the pinning effect, we devised a composition that reduces the δ ferrite temperature range, where the rate of Ti diffusion is high and TiN tends to coarsen.[33)] We also developed a technique that further improves HAZ toughness by reducing coarse TiN formed because of oxides. Specifically, the technique involves modifying oxides by adding Ca.[34)]

One method of refining the microstructure by transformation during cooling is to disperse particles that can become transformation nuclei within the austenite grains and form the desired microstructure from within the grains. We used this method to develop steel plate that is compatible with high-heat-input welding by dispersing BN as intragranular nucleation sites.[35)] We also developed a HAZ microstructure refinement technique that uses a composite oxide of Ti, rare earth elements (REM), and Zr.[36)]

Such intragranular nucleation techniques are actively used in weld metals with a high oxide content. For example, in weld metal for high-strength steel, $MnTi_2O_4$[37), 38)] is used as an intragranular nucleation site for microstructure refinement, resulting in weld metal with excellent toughness. It has also been found that adding Li to the weld metal further improves its toughness and enhances microstructure refinement.[39)]

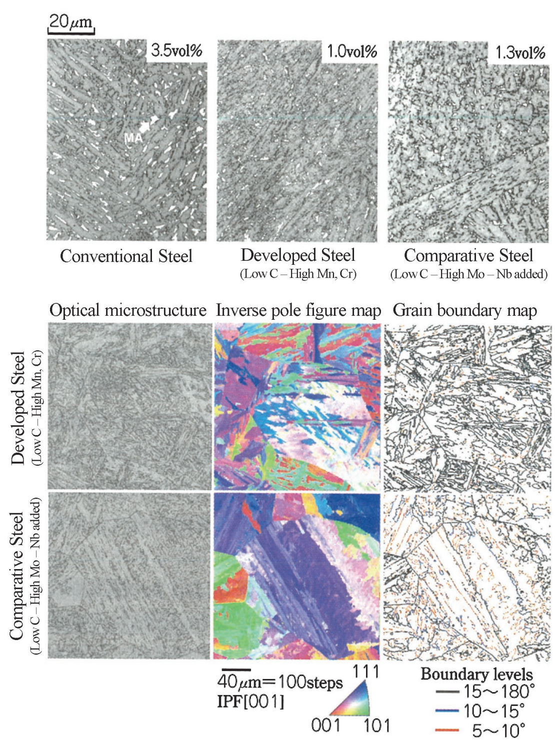

Fig. 4 Optical microstructure and EBSD analysis results of simulated HAZ (heat-affected zone)

As described in this section, we have been developing metallographic structure control technology for both steel and welding consumables to efficiently implement safe and secure community development. These objectives are met because our developments increase the strength of materials used in large buildings, bridges, and other structures and ensure toughness in high-heat-input welding. Structures built during a period of rapid economic growth have begun deteriorating in recent years, indicating a need to ensure the safety of societal infrastructure for the next 50 to 100 years. We are pursuing technological development to provide the properties necessary for long-term reliability, such as fatigue resistance and corrosion resistance on top of the conventionally sought property of toughness.

3. Prospects of metallographic structure control

As described, new developments in metal materials are being pursued to overcome various challenges. However, material performance demands have been increasing, with the challenges of development increasing in tandem. Societal change is likewise accelerating, necessitating faster development of new metal materials. One solution is to enhance microstructure prediction and property prediction techniques. These support calculations related to atomic phenomena that govern the formation of metallographic structures and their resulting material properties.[40), 41), 42), 43)] Such information deepens the understanding of metallurgical phenomena and enables quantitative estimation of metallographic structures and material properties resulting from composition and the processes used. This in turn promotes materials development methodology that makes use of computational science and the knowledge and experience of engineers.

Furthermore, the recent trend toward digital transformation (DX) has fostered interest in a technical field known as MI to bring about DX in materials technology. MI can stand for *materials informatics*, which refers to technology that aids in materials development by using information science tools such as machine learning. Alternatively, MI can stand for *materials integration*, referring to technology that aids in materials development by combining materials science theory, experimentation, analysis, simulation, databases, data mining, etc. It is difficult to collect large amounts of experimental data in the field of metal materials. As such, it is challenging to develop purely data-driven MI techniques, and it is useful to combine MI with the prediction techniques developed. As an example, our development assistance technology combines machine learning with technology for predicting toughness based on the behavior of microstructure formation and the microstructure itself in the HAZ of steel plate. Our development assistance technology can also present compositions that satisfy stipulated properties, alloy cost, etc. for a given welding condition.[44] To advance such technology, information related to material properties must be quantified so that it can be used for data analysis. We will advance metallographic structure control technology by linking it with related fundamental technologies. These include quantitative technologies such as microstructure imaging for material properties as well as atomic-level analysis and evaluation technology.

Conclusions

As described above, we have continuously developed metallographic structure control technology to contribute a safe, secure, and sustainable society. This paper has covered the microstructure control technologies targeted at reducing the weight of vehicle bodies and ensuring the safety factor of large structures. However, these technologies are used in a wide range of applications, including reducing the weight of other transportation equipment such as aircraft (titanium, aluminum) and ships (steel plate, welding consumables, cast and forged steel), improving the efficiency of internal combustion engines in vehicles (wire rod and bar), improving power generation efficiency in the energy industry (steel plate, welding consumables), and reducing CO_2 by streamlining the manufacturing processes of products that use various metals.

While we will continue refining our existing technologies to solve today's challenges, we will also develop new technologies to address major anticipated societal changes. Such changes will subject metal materials to unprecedented environments and use cases, meaning that related developments must occur via a multi-pronged approach. Anticipated changes include the expanded use of hydrogen, ammonia, and renewable energy in support of carbon neutrality, the shift away from fossil fuels in transportation equipment as exemplified by the shift to electric and fuel cell vehicles, the implementation of next-generation air mobility, and the advancement of the space industry. Necessary material properties will be defined from new perspectives, such as performance in the extreme environments of cryogenic and ultra-high temperatures, the addition of new functions including electromagnetic properties, and the realization of unprecedented specific strength and specific rigidity. By continuing to develop and provide metal materials with performance characteristics that can live up to such changing challenges, we are advancing metallographic structure control technology in support of safe, secure, and sustainable societal infrastructure.

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