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Technical Review

Product Development for Functional Coated Iron-based Metal Foils

Hiroto UNNO*
Yoshiaki HAGIWARA
Jun NAKATSUKA
Toru SAITO
Taku KAGAWA

Abstract

In addition to surface finishes using foil rolling technology, which is one of the strengths of Nippon Steel Chemical & Material Co., Ltd., an iron-based metal foil with a functional film has been developed in which the surface is coated with a film consisting of a siloxane network modified with an organic group. The surface treatment technology makes it possible to impart new functions such as heat resistance, barrier properties, and planarization to the metal foils. Taking advantages of these functions, products are expected to be developed in new applications using semiconductor processes, such as current collectors for oxide-based thin-film all-solid-state batteries and high-temperature superconducting coil substrates for fusion reactors.

1. Introduction

Stainless steel foils are cold rolled to a thickness of 100 μm or less. They are widely used in the structural components of exhaust catalyst carriers and hard disk drives and the spring components of switches due to their excellent mechanical properties.[1] Nippon Steel Chemical & Material Co., Ltd. has an integrated production and quality control system, from raw material procurement to foil rolling, within the Nippon Steel Group and has strengths in rolling technology that can control extremely thin thickness with high precision while achieving high flatness. We hold a significant global share in the application of stainless steel foils for hard disk drive suspensions. In recent years, with the progress of miniaturization and high performance of devices, various high-performance requirements have been specified for iron-based metal foils, including stainless steel, according to the application. Therefore, we have been considering the addition of new functionality by composites with resin films[2] and surface treatments[3] and the development of products that utilize these elemental technologies. In this report, we describe the characteristics of stainless steel foils manufactured by Nippon Steel Chemical & Material, introduce the surface treatment by using the sol-gel technology, and outline the product development trends of functional film-coated iron-based metal foils using the sol-gel surface treatment technology.

2. Features of Stainless Steel Foil Made by Nippon Steel Chemical & Material

The general manufacturing process of stainless steel is roughly divided into the following four processes: (1) electric furnace to continuous casting steelmaking process, (2) hot rolling process, (3) cold rolling process including cold rolling and annealing and pickling, and (4) finishing process. In the foil rolling process (3), stainless steel has the highest hardness compared to commonly used materials such as aluminum, copper, and nickel (Ni) alloys. Therefore, a multi-stage cluster rolling mill with small diameter work rolls is used due to the constraints on the rolling load. At Nippon Steel Chemical & Material, a 12-stage rolling mill is used to roll ultra-thin stainless steel foils with high precision. An excellent thickness accuracy of ± 0.3 μm can be accomplished even for ultra-thin foils with a thickness of 10 μm. In addition, we have continuous degreasing and cleaning equipment and a bright annealing furnace. Ultra-thin stainless steel foils are transported at high speed so as not to cause deflection. We can thus achieve high productivity and temper rolling, enabling a wide range of mechanical properties, as shown in **Table 1**. In addition, the surface finishing cultivated in suspension applications is a significant feature. Various surface finishes can be produced, as shown in **Table 2**. The relationship between the surface roughness Ra of stainless steel foils and the specular glossiness of GS 45° is shown in **Fig. 1**. Generally, the specular glossiness in-

* PhD., Group Leader, New Materials Development Center, Research & Development Div., Nippon Steel Chemical & Material Co., Ltd.
1 Tsukiji, Kisarazu City, Chiba Pref. 292-0835

Table 1 Production grades

Characteristics		Symbol of grade	Typical composition	Applications	Chemical composition by selected element (weight %)									
					C	Si	Mn	P	S	Ni	Cr	Mo	N	Other significant elements
Stainless steel Austenitic type	Strain hardening	SUS301	17Cr-7Ni	Tact switch Disk springs	≦0.15	≦1.00	≦2.00	≦0.045	≦0.030	6.00～8.00	16.00 ～ 18.00	—	—	—
	General use	SUS304	18Cr-8Ni	HDD suspension Battery case	≦0.08	≦1.00	≦2.00	≦0.045	≦0.030	8.00～10.50	18.00 ～ 20.00	—	—	—
	Corrosion resistance	SUS316L	18Cr-12Ni-2.5Mo-LC	Low-magnetic springs	≦0.030	≦1.00	≦2.00	≦0.045	≦0.030	12.00～ 15.00	16.00～ 18.00	2.00～3.00	—	—
Stainless steel Ferritic type	Formability	SUS430LX	17Cr-0.4Ti-LC,N	Battery current collectors	≦0.030	≦0.75	≦1.00	≦0.040	≦0.030	—	16.00～ 19.00	—	—	Ti or Nb 0.10～1.00
	Corrosion resistance	SUS444	19Cr-2Mo-Nb,Zr-LC,N	Battery current collectors	≦0.025	≦1.00	≦1.00	≦0.040	≦0.030	—	17.00～ 20.00	1.75～ 2.50	≦0.025	—
	High temperature Oxidation resistance	YUS205-M1	20Cr-5Al	Automotive catalytic converter Film heater	≦0.010	≦0.50	0.15 ～ 0.25	≦0.035	≦0.005	—	19.5 ～ 20.5	—	≦0.010	Al：4.8～5.2
Nickel-coated steel	Corrosion resistance Low resistance	SUPERNICKEL	Nickel-coated low carbon steel	Battery current collectors	≦0.13	≦0.04	≦0.60	≦0.035	≦0.05	—	—	—	—	—

Characteristics		Symbol of grade	Symbol of thermal refining	Mechanical properties (Reference values)				Physical properties						
				Tensile strength N/mm^2	Proof stress N/mm^2	Elongation %	Hardness HV	Density g/cm3 (RT)	Young's modulus kN/mm2 (0～100°C)	Specific heat kJ/kg/°C (0～100°C)	Thermal conductivity W/ (m°C) (100°C)	Coefficent of thermal expansion $\times 10^{-6}$/°C (100°C)	Electric resistivity $10^{-8}\Omega m$ (RT)	Amplitude permiability μ Peduction (0/50%)
Stainless steel Austenitic type	Strain hardening	SUS301	H-TA	1430	1230	26	440	7.93	193	0.50	16.3	16.9	72	—
			EH-TA	1810	—	—	550							
			SEH-TA	1910	—	—	570							
	General use	SUS304	BA	820	460	39	250	7.93	193	0.50	16.3	17.3	72	1.000/2.0
			H	1360	1280	1	400							
			H-TA	1460	1370	1	460							
	Corrosion resistance	SUS316L	BA	650	330	31	180	7.98	193	0.50	16.3	16.0	74	1.004/1.027
			H	1230	1180	2	390							
Stainless steel Ferritic type	Formability	SUS430LX	H	880	—	—	270	7.7	200	0.50	0.5	26.0	60	—
	Corrosion resistance	SUS444	BA	680	600	17	230	7.7	200	0.46	0.46	36.9	—	—
			H	1040	—	—	300							
	High temperature Oxidation resistance	YUS205-M1	BA	680	570	21	230	7.2	169	0.46	11.7	11.5	135	—
			EH	1380	—	—	410							
Nickel-coated steel	Corrosion resistance Low resistance	SUPERNICKEL	H	750	720	1	—	7.85	—	—	—	—	—	—

creases with the decrease in surface roughness. To achieve the low specular glossiness required for this application, we developed a MILK WHITE™ (hereinafter abbreviated to MW) finish with a small difference in the specular glossiness between the rolling direction and the width direction. The MW finish stainless steel foil has become the de facto industry standard material.

3. Surface Treatment Using Sol-Gel Technology[*1]

We have been studying how to provide a flat surface, barrier properties, and electrical insulation by coating the surface of stainless steel foils with a film made of a siloxane skeleton modified with an organic group.[3)] The Advanced Technology Research Center of

[*1] Sol-gel technology: A material technology that uses a solution as a starting material to create organic-inorganic hybrids, glasses, ceramics, etc.

Table 2 Surface finishes

Surface: Finishes	Specular Glossiness GS (45°)	Roughness Ra	Roughness Rmax
	%	μm	μm
MILK WHITE™	400	0.05	0.60
Super Bright	800	0.02	0.20
Bright A	600	0.03	0.35
Bright D	200	0.60	5.00

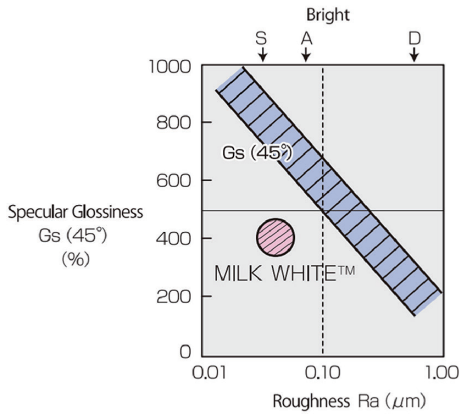


Fig. 1 Relation between specular glossiness and roughness

Nippon Steel Corporation designed the coating solution that meets these required characteristics. Details will be given in another report. In anticipation of the mass production of iron-based metal foil products with functional films, Nippon Steel Chemical & Material introduced a roll-to-roll system for continuous processing. The main processes consist of (1) a corona treatment process required for wettability modification of the foil substrate surface, (2) a coating and drying process in which a thin film is formed using the coating solution, and (3) a heat treatment process in which a dehydration condensation reaction forms a hardened film. The corona treatment process (1) is a treatment technology that modifies the substrate surface by corona discharge oscillating from a high-frequency power supply device. The polar functional groups introduced into the substrate surface improve the wettability, enabling uniform coating. Water contact angle measurements confirmed that corona treatment reduced the angle to less than 10° for SUS304 (20 μm, H-TA, MW finish) and SUS444 (10 μm, H, Bright D finish) foil strips. The outline of the roll-to-roll equipment used in the coating and drying steps of the process (2) is shown in **Fig. 2**. A coater with a coating width of 500 mm was installed. A metering pump capable of steadily supplying the solution even at a low flow rate was adopted to form a uniformly thin film. The drying furnace is a hot air circulation type. The outline of the roll-to-roll equipment used in the heat treatment step of the process (3) is shown in **Fig. 3**. A far-infrared heating furnace capable of uniform high-temperature heat treatment within the surface was adopted. The maximum operating temperature is 420°C. The roll-to-roll equipment can transport substrates up to 540 mm wide and can handle various substrates such as copper foils and plastic films in addition to stainless steel foils.

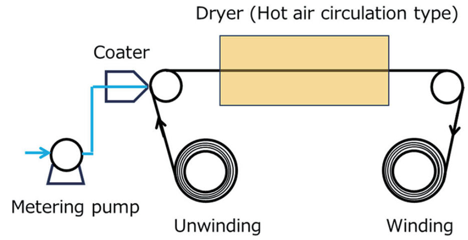


Fig. 2 Schematic diagram of Roll-to-Roll coating and drying equipment

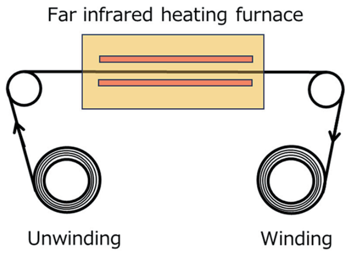


Fig. 3 Schematic diagram of Roll-to-Roll heat curing equipment

4. Product Development for Oxide-Based Thin-Film All-Solid-State Batteries

All-solid-state lithium-ion secondary batteries are attracting attention as next-generation high-performance storage batteries that combine high energy density and safety. Such batteries are made of solid materials for the positive and negative electrodes and electrolytes. They are characterized by high safety, with no risk of leakage or fire. However, the manufacturing process and the environment in which the battery's constituent materials are placed are significantly different from those of conventional organic electrolytes. In particular, oxide-based all-solid-state lithium-ion secondary batteries using oxide-based solid electrolytes that are stable even in the air are suitable for wearable devices in which safety is important. If they are made of thin films, they can contribute to miniaturization, weight reduction, and shape flexibility. The structure of a typical oxide-based thin-film all-solid-state lithium-ion secondary battery is shown in **Fig. 4**.[4] Platinum (Pt) is the positive electrode current collector and is formed on a Si wafer substrate covered with SiO_2. The battery device layer and encapsulation material are laminated on top of the Pt layer. Dry film deposition technology, which is more suitable for thin film deposition than conventional wet film deposition and is used in many semiconductor manufacturing processes, is applied to each constituent layer of the battery device.[4] However, lithium cobalt oxide (LCO), a typical positive electrode material, has

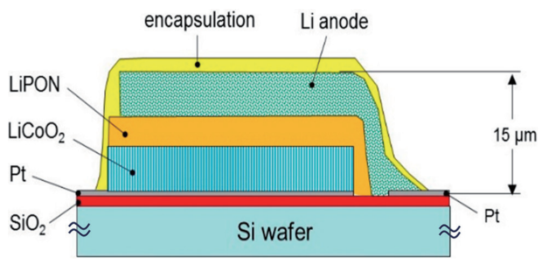
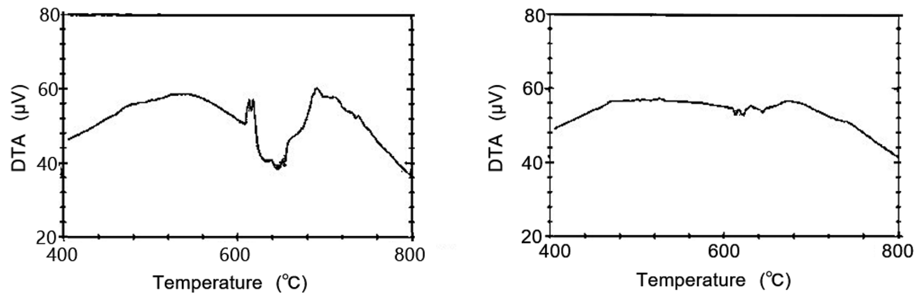


Fig. 4 Schematic cross-section illustrating the layout of an all-solid-state thin-film battery



(a) Aluminum foil (b) SUS444 stainless steel foil

Fig. 5 Differential thermal analysis results of LCO coated (a) aluminum foil and (b) SUS444 stainless steel foil

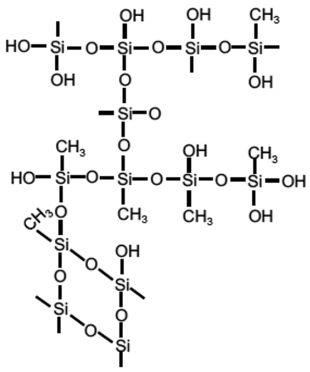


Fig. 6 Schematic of silica-based coatings

low crystallinity immediately after dry deposition and requires crystallization heat treatment at 600°C or higher to improve the energy capacity.[4] Therefore, when conventional aluminum (Al) foil is used as a positive electrode current collector that holds the LCO, its heat resistance becomes an issue because the melting point of Al is low at about 660°C. **Figure 5** shows the results of the differential thermal analysis of the LCO-positive electrode material deposited on the Al foil and stainless steel foil in a nitrogen gas flow environment.[5,6] No significant thermal reaction was observed on the stainless steel foil even when heated to 700°C. An exothermic reaction occurred on the Al foil before the melting heat absorption occurred at 600°C or higher. This reaction is thought to be due to the thermite reaction in which oxygen in the positive electrode material reacts with the Al in an inert gas environment. Stainless steel has a high melting point of about 1 400°C and has excellent heat resistance, so it can be used as a current collector for thin-film batteries in which dry deposition is applied. Furthermore, depending on the battery structure, the stainless steel foil itself may be exposed to a dry film formation environment. It is predicted that the surface composition of the stainless steel foil will change due to surface oxidation, etc. Therefore, Nippon Steel Chemical & Material has developed a stainless steel foil with a functional silica-based film (**Fig. 6**) containing a certain amount of organic components as a barrier film to reduce environmental impact.[3] Generally, silica-based films become embrittled due to the condensation polymerization caused by heat treatment, and cracks occur due to the stress associated with volume shrinkage. However, this problem has been solved by the unique composition design containing a certain amount of organic components and heat treatment technology. The film is expected to be used as a current collector for oxide-based thin-film all-solid-state lithium-ion secondary batteries.

5. Development of Stainless Steel Foil with Functional Film for High Heat Resistance Applications

Based on the achievements of oxide-based thin-film all-solid-state lithium-ion secondary batteries, we are working to improve the functionality of functional films, such as higher heat resistance, flatness, and insulation, and to study their use in new applications such as functional devices using semiconductor manufacturing processes. One of the candidates is a component for nuclear fusion reactors, which are expected to be a dream power generation technology. In the most traditional method of tokamak nuclear fusion power generation, ultra-high temperature and high-pressure plasma is confined in a doughnut-shaped magnetic field generated by a coil to cause nuclear fusion. In the International Thermonuclear Experimental Reactor project launched in 1978, low-temperature superconducting coils were used for the superconducting coils that generate the magnetic field, and the large size of the reactor and economic aspects prevented its early practical application.[7] However, in recent years, it has been found that the use of high-temperature superconducting (HTS) coils with a strong magnetic field can reduce the size of the reactor and construction costs, so commercial reactors are expected to be put into practical use in the mid-2030s. Currently, this HTS coil is manufactured by forming a three-dimensionally oriented ceramic thin film as an intermediate layer on a highly flat heat-resistant Ni alloy substrate such as Hastelloy using ion beam assisted deposition and then forming a superconducting thin film using metal-organic deposition or metal-organic chemical vapor deposition (MOCVD).[8,9] Therefore, the surface roughness and flatness of the substrate, which affect the formation of the intermediate layer and superconducting layer, as well as the orientation and crystallinity of the crystal grains, greatly affect the performance of the HTS coil. In addition, heat resistance is also required because the process temperature of the MOCVD method reaches nearly 900°C.[8,9] At Nippon Steel Chemical & Material, the above-mentioned surface treatment technology allows the formation of a coating with relatively high productivity. In addition to designing the coating to suit specific applications, we also provide various methods, such as texture control by rolling and steel inclusion control. We will accelerate the practical application of functional film-coated iron-based metal foils by combining the technologies mentioned above and proposing them to customers.

6. Summary and Outlook

Nippon Steel Chemical & Material has been developing products by taking advantage of the strengths of the integrated production and quality control system within the Nippon Steel Group, rolling technology that can realize high thickness accuracy and flatness, and surface finishing, including MW finishing with a small difference in specular glossiness between the rolling direction and the width direction. In the future, with the progress of surface treatment using sol-gel technology, products are expected to be developed for new applications, such as functional devices using semiconductor manufacturing processes by improving the functionality of iron-based metal foils, such as higher heat resistance, higher flatness, and higher insulation.

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Hiroto UNNO
PhD., Group Leader
New Materials Development Center
Research & Development Div.
Nippon Steel Chemical & Material Co., Ltd.
1 Tsukiji, Kisarazu City, Chiba Pref. 292-0835

Toru SAITO
PhD., Group Leader
Process Development Center
Research & Development Div.
Nippon Steel Chemical & Material Co., Ltd.

Yoshiaki HAGIWARA
Ph.D., Chief Manager, Head of Section
New Materials Research Lab.
Advanced Technology Research Laboratories
Nippon Steel Corporation

Taku KAGAWA
Researcher
New Materials Development Center
Research & Development Div.
Nippon Steel Chemical & Material Co., Ltd.

Jun NAKATSUKA
Business Development Planning Dept.
Nippon Steel Chemical & Material Co., Ltd.