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Synopsis :

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Production of Deep Drawing Quality Steel Sheets for Porcelain Enameling by Continuous Casting*[1]

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A deep drawable cold rolled steel sheet for enameling use was developed. The steel sheet was made from continuously cast Ti bearing steel with extremely low C content. Processing conditions of the steel sheet are discussed in this paper. Fishscaling is more effectively prevented by the use of TiN precipitates rather than TiC precipitates. TiN in steels less deteriorates press formability under any hot rolling condition than TiC. Smut deposited on the steel surface increases during pickling for enameling pretreatment with increasing Ti content in steel, resulting in poor enamel adhesion. The amount of Ti in steel must be restricted to less than 0.06% to obtain excellent enamel adhesion. Continuously annealing process is preferable for preventing cold-work embrittlement in the steel.

1 Introduction

To obtain beautiful porcelain enamel surfaces and good porcelain enamel adhesion, cold-rolled enameling steel sheets are required to provide various enameling properties in addition to the properties required of general cold-rolled steel sheets. That is to say, it is necessary to minimize the carbon in steel in order to prevent porcelain enamel defects, such as blistering and pinholes, and reduce warping; it is also necessary to increase the hydrogen-occluding capacity of steel sheets by properly dispersing secondary phases, such as inclusions and precipitates, into steel in order to prevent the occurrence of fishscale.[1] Furthermore, the steel sheets must have excellent press formability because many porcelain enameled products are formed by severe press forming as with bath tubs and kitchen appliances.

Steel sheets produced by decarburizing annealing after the hot and cold rolling of capped steel ingots have been used as enameling steels that provide these various properties.[1] Capped steel includes many oxide inclusions and fish scale scarcely occurs. In addition, pinholes and warping can be prevented if the carbon content of capped steel is lowered to extra-low levels by decarburizing annealing. However, the slabbing process is necessary because capped steel is produced by ingot casting. Moreover, cold-rolled steel sheets of capped steel show great variations in the mechanical properties and porcelain enameling properties within the same coil because sulfur, phosphorus, oxygen, etc., segregate in the ingot during casting. To obtain extra-low-carbon steels, it is necessary to conduct decarburizing annealing at high temperatures for a long time and this leads to high production cost. The thickness of steel sheets must be 0.6 mm or more in order to obtain good sheet flatness.

If extra-low-carbon steels produced by vacuum degassing in the molten steel stage are continuously cast, variations in the properties of products are small, and it is unnecessary to conduct decarburization during annealing. Therefore, it is possible to conduct batch annealing or continuous annealing, and steel sheets of good flatness can be produced without thickness limitations. However, it is difficult to decarburize steel by vacuum degassing to carbon contents of the same degree as with decarburizing annealing, and this steel is inferior to decarburized capped steel in the mechanical properties, especially aging property. Moreover, if high-oxygen steels such as capped steel are continuously cast, the carbon and oxygen in steel react, generating blow-

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holes on the slab surface. Therefore, good steel surfaces cannot be obtained. The deterioration in the mechanical properties is small if the carbon remaining in steel is fixed by carbide formers such as zirconium, titanium and niobium. To add these carbide formers to steel, however, it is necessary to remove the oxygen in steel beforehand. Deoxidation is necessary also for obtaining good steel surfaces as it prevents blowholes. On the other hand, deoxidation reduces oxide inclusions in steel; therefore, it is necessary to take measures as an alternative for oxide inclusions to prevent fishscale. In steels to which titanium, zirconium, niobium, etc., are added, the carbon remaining in steel is dispersed as carbides; these carbides increase the hydrogen-occluding capacity of steel sheets in place of oxides, and hence, fishscale is less apt to occur.[2)] The deterioration in the mechanical properties, especially ductility is unavoidable, however, if carbides are dispersed in steel in amounts capable of completely suppressing fishscale. To produce enameling steel sheets by this method, therefore, it is necessary to rigidly control the composition of steel, especially the carbon content and the amounts of carbide formers, such as titanium, niobium, and zirconium. Therefore, it has so far been virtually impossible to produce steel sheets that combine excellent fishscale resistance and press formability.

To simultaneously meet the fishscale resistance and press formability which are incompatible with each other, fishscale resistance was improved by enriching nitrides in place of carbides using Ti-bearing steels, and enameling steel sheets excellent in both fishscale resistance and press formability were developed. This paper describes manufacturing conditions of these enameling steel sheets and presents their mechanical properties and porcelain enameling properties.

Table 1 Chemical composition of steel

(wt%)

C	Si	Mn	P	S	N	Ti
0.002 ~ 0.008	0.01 ~ 0.03	0.05 ~ 0.09	0.010 ~ 0.015	0.005 ~ 0.015	0.002 ~ 0.008	0.05 ~ 0.11

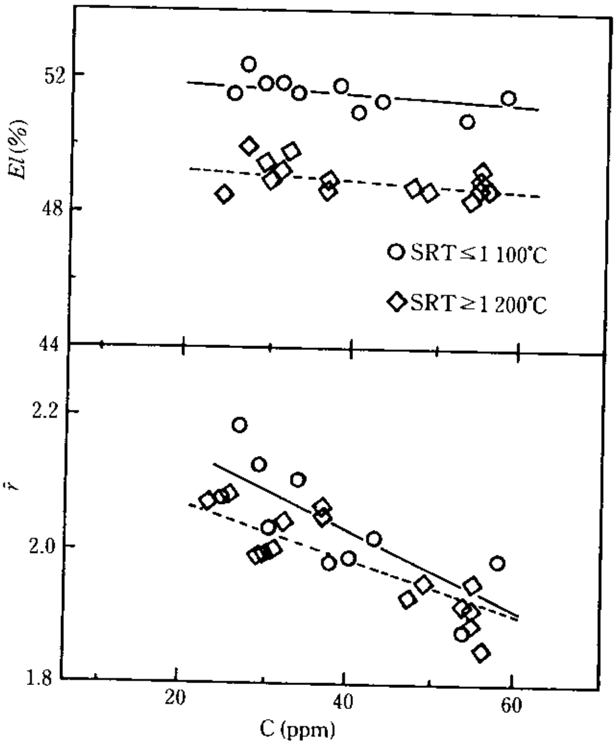


Fig. 1 Influence of C on total elongation and $\bar{r}$ value of cold rolled steel sheet

2 Effects of Hot Rolling Conditions on Mechanical Properties of Ti-Bearing Steels

Steels of the chemical composition given in **Table 1** were made by way of trial by converter refining, vacuum degassing and continuous casting to investigate effects of hot rolling conditions on the press formability of Ti-bearing steels. Slabs were hot rolled to 2.8-mm thick sheets by varying the slab-reheating temperatures (SRT) between 1 000 and 1 300°C, the finisher delivery temperatures (FDT) between 750 and 880°C and the coiling temperatures (CT) between 550 and 700°C and were then cold rolled to 0.7-mm thick sheets. Subsequently the cold-rolled sheets were continuously annealed at 800 ± 20°C for 30 s of soaking and temper-rolled with a rolling reduction of 0.7%. After that, the tensile properties and $\bar{r}$-value were investigated.

Ductility (*El*) and $\bar{r}$-value are most related to deep-drawability among the mechanical properties of cold-rolled steel sheets. **Figure 1** shows the effect of the carbon content on these values when the slab-reheating temperature is 1 100°C or below and when it is 1 200°C or above. The effect of the SRT on ductility is stronger than that of the carbon content when $C < 70$ ppm; *El* is 50% or higher at low SRT's, and 48% or higher at high SRT's. On the other hand, the $\bar{r}$-value does not depend on the SRT and decreases with increasing carbon content; $\bar{r} \geqq 2.0$ at $C \leqq 30$ ppm and $\bar{r} > 1.8$ at $C = 30$ to 50 ppm. Similary, **Fig. 2** shows the effect of the finisher delivery temperature on *El* and $\bar{r}$-value. Both *El* and $\bar{r}$-value decrease a little with decreasing FDT. However, the degree of this deterioration depends on the SRT. That is to say, *El* and the $\bar{r}$-value are better at low SRT's than at high SRT's and $El > 50\%$ and $\bar{r} > 1.8$ can be obtained even at FDT of 750°C. When the SRT is low, it is difficult to hold the FDT at a high level. However, the same mechanical properties as with high SRT's and high FDT's of 850°C or above can be obtained by a low FDT

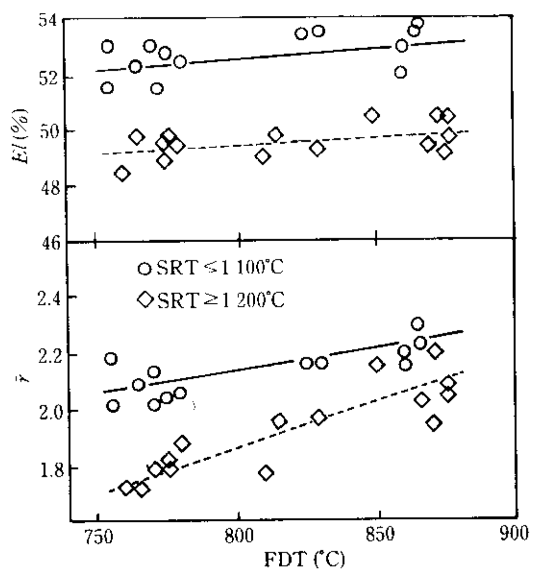


Fig. 2 Influence of FDT on total elongation and $\bar{r}$ value of cold rolled sheet steel

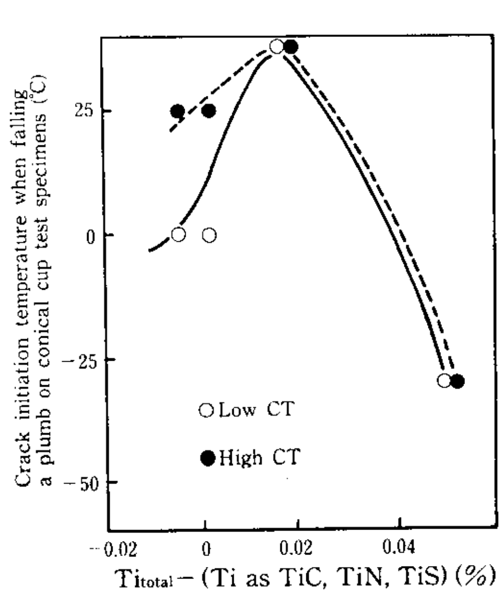


Fig. 3 Influence of Ti content in steel on stiffness of sheet after pressforming

of 750°C. The effect of the coiling temperature on the mechanical properties of cold-rolled steel sheets is smaller than those of the SRT and FDT, and it is appropriate to coil the strip at temperatures of 600°C or below that facilitate the descaling before cold rolling.

3 Effects of Ti Content of Steel and Annealing Method on Secondary Working Embrittlement

It is known that when the amounts of solute carbon and nitrogen in steel become very minute, grain boundaries become brittle with the result that pressed products fracture under a small external stress. This is what is called secondary working embrittlement. One of the causes of this embrittlement is said to be the boundary segregation of phosphorus.[3] Secondary working embrittlement is apt to occur in Ti-bearing steels because carbon and nitrogen are fixed as TiC and TiN, respectively.

Steel sheets with different Ti contents were box-annealed at 720°C for 5 h and invesigation was made into the effect of the Ti content of steel on secondary working embrittlement. **Figure 3** shows results of this investigation. The embrittlement test was made by dropping a 5-kg plumb from a height of 2 m on test pieces formed into conical cups at various temperatures, to see whether cracks occurred or not.

The most noticeable embrittlement is observed when the remaining Ti content after the subtraction of Ti as TiC, TiN, and TiS from the Ti in steel is about 0.02%. Embrittlement improves at lower or higher Ti contents than this value. Especially when the remaining Ti content is 0.04% or more, secondary working embrittlement is conspicuously suppressed. When the remaining Ti content is 0.02% or less, the improvement in brittleness is not clear at high coiling temperatures in the hot rolling process and embrittlement is somewhat improved by low-temperature coiling.

Table 2 shows amounts of phosphorus present in steel as precipitates after annealing, together with the contents of other elements. In steels with high Ti contents, phosphorus precipitates are present and solute phosphorus is notably small in quantity. These precipitates were extracted by X-ray diffraction and the extraction replica method and the extracted precipitates were analyzed by the EDS (Energy dispersion spectroscopy) under a transmission electron microscope. As a result, they were identified as TiFeP.

It is apparent from these results that addition of a sufficient amount of titanium is necessary for fixing phosphorus as precipitaes in order to prevent secondary working embrittlement in the case of box annealing. As will be described later, however, porcelain enamel adhesion decreases with increasing Ti content of steel; therefore, this method is not suitable for the production of enameling steel sheets.

Figure 4 shows a comparison of brittleness when steel sheets of different Ti contents were box-annealed or continuously annealed. The continuous annealing temperature is 800°C. In the steel sheet of 0.06% Ti, cracks are formed even at 0°C when this steel sheet is produced by box annealing. In the case of continuous annealing, however, cracks are scarcely formed down to −60°C as with the steel sheets of 0.06% Ti. This seems to be because in the case of continuous annealing, the bound-

Table 2 Change in solute P (wt %)

Steel	CT	C	S	N	Ti	P_{total}[1)]	P_{ppt}[2)]	P_{sol}[3)]
1	550	0.002	0.009	0.0035	0.021	0.010	<0.0003	0.010
	700	0.002	0.009	0.0035	0.021	0.010	<0.0003	0.010
2	550	0.004	0.009	0.0039	0.025	0.010	<0.0003	0.010
	700	0.004	0.009	0.0039	0.025	0.010	<0.0003	0.010
3	550	0.004	0.001	0.0042	0.043	0.008	<0.0003	0.008
	700	0.004	0.001	0.0042	0.043	0.008	0.0003	0.008
4	550	0.006	0.009	0.0039	0.094	0.008	0.007	0.0009
	700	0.006	0.009	0.0039	0.094	0.008	0.007	0.0009

1) P_{total} : total P
2) P_{ppt} : preciptated P
3) P_{sol} : solute P

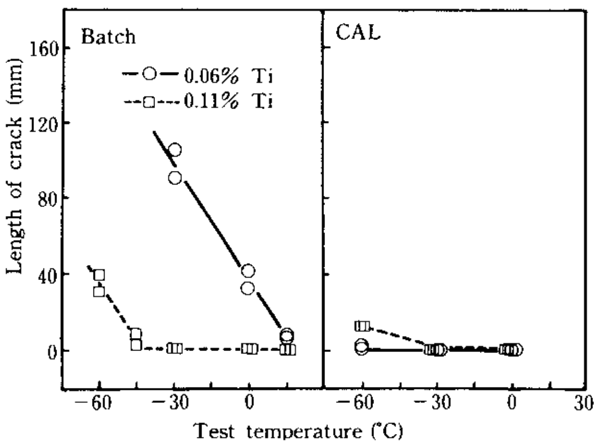


Fig. 4 Change in stiffness of steel sheet after press-forming according to Ti content and annealing method

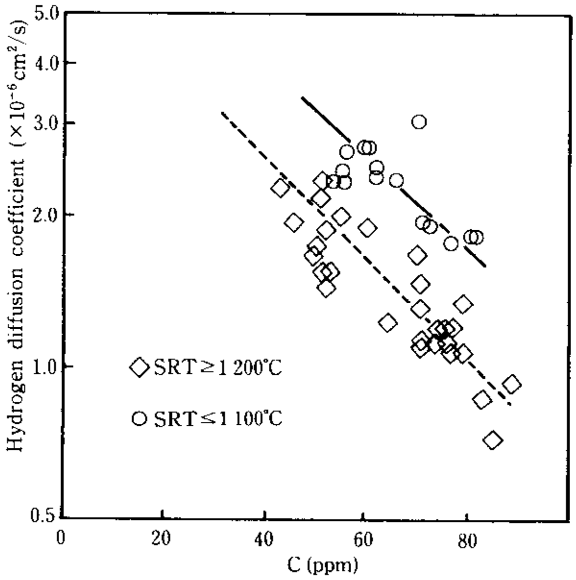


Fig. 5 Influence of C on apparent hydrogen diffusivity

ary segregation of phosphorus hardly tends to occur due to high cooling rates of steel sheets. Therefore, continuous annealing is necessary for preventing the secondary working embrittlement of steel sheets of low Ti contents.

4 Effects of Steel Compositions and Hot Rolling Conditions on Porcelain Enameling Properties

4.1 Effects of C Content in Steel and Slab-Reheating Temperature on Fishscale Resistance

It is known that the fishscale resistance of Ti-bearing steels depends strongly on the amount and distribution of TiC precipitates in steel.[2)] **Figure 5** shows the effect of the C content of steel on the apparent diffusion coefficient (*D*), which is considered to correspond with the fishscale resistance of steel sheets. *D* was determined by measuring the change with time in the amount of hydrogen liberated from one side of the steel sheet when hydrogen was charged from the other side by cathodic electrolysis.[1)] *D* decreases with increasing C content. When the C content is the same, *D* is smaller at high SRT's than at low SRT's. Therefore, it might be thought that fishscale resistance is good at high C contents and high SRT's as *D* is small under these conditions. **Figure 6** shows the tendency toward fishscaling in actually porcelain enameled steel sheets rearranged in terms of the C content of steel and SRT. Pretreatment for the porcelain enameling was only a pickling with 10% H_2SO_4 aq. solution (75°C) for 20 s, using the L type ground coat slip made by Nippon Ferro Co., Ltd. (standard firing tem-

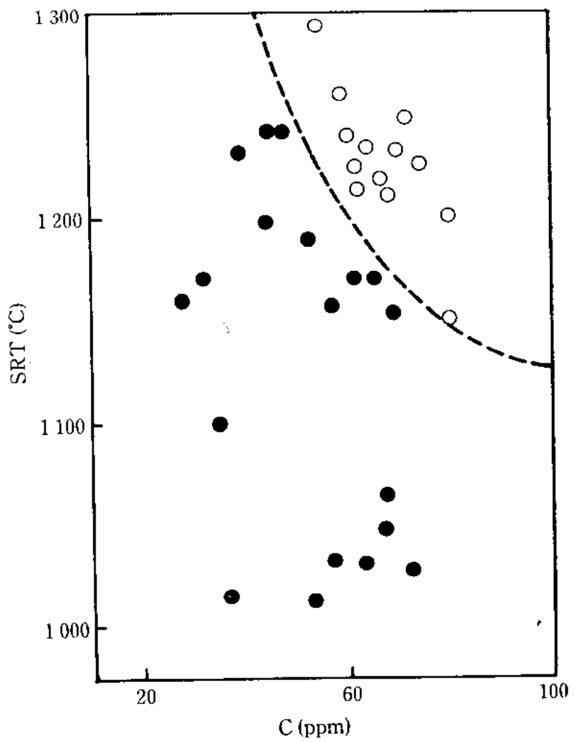


Fig. 6 Condition in chemical composition and hot rolling to avoid fishscaling (●: fishscaling ○: fishscale free)

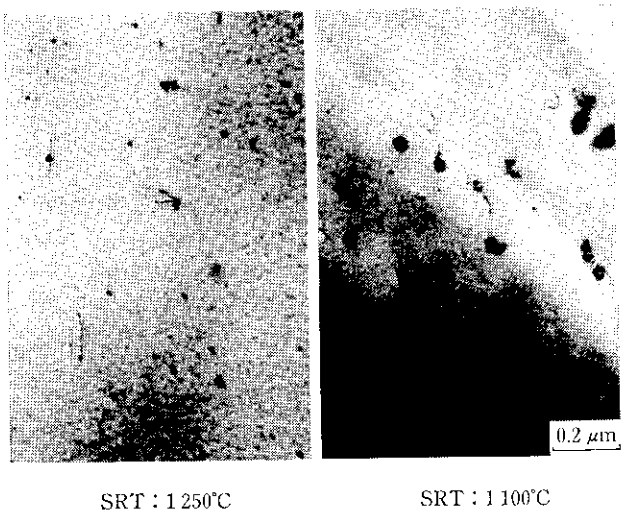


Photo 1 Influence of SRT on TiC dispersion

perature: 820°C). The existence of fishscale was examined after the treatment for promoting fishscaling at 160°C for 16 h. The higher the SRT and the higher the C content of steel, the less fishscale tends to occur. It is apparent from Fig. 5 that D-values of 1.5×10^{-6} cm^2/s or less are desirable. **Photo 1** shows a comparison of the distribution of TiC in steel between a high-SRT steel and a low-SRT steel. The TiC precipitates in the steel reheated to an SRT of 1 100°C are coarser than those in the steel reheated to 1 250°C. It is considered that the former steel has higher D for this reason and is more susceptible to fishscaling.

4.2 Effect of N Content of Steel on Fishscale Resistance

Titanium is an element that easily forms not only carbides but also nitrides and sulfides in steel. As mentioned above, to prevent fishscale by utilizing carbides, it is necessary to increase the C content of steel and to raise the SRT, but this makes it difficult to avoid the deterioration in press formability. Therefore, the effect of nitrides on fishscale resistance was investigated by making a comparison with a steel of increased C content. The three types of steel with different chemical compositions shown in **Table 3** were used in the experiment. Steel A is an extra-low-carbon extra-low-nitrogen steel, steel B a carbon-added extra-low-nitrogen steel, and steel C an extra-low-carbon nitrogen-added steel. To compare the tendency toward fishscaling, steel sheets were fired at three different dew points of 20, 30, and 40°C and the condition of fishscaling was investigated. Pickling was conducted for 20 sec as porcelain enameling pretreatment; other enameling conditions are the same as those described above. **Table 4** shows the number of sheets in which fishscale occurred when three sheets were fired in each case. In steel A, fishscale

Table 3 Chemical composition of steels

(wt%)

Steel	C	Si	Mn	P	S	N	Ti
A	0.0030	0.016	0.09	0.012	0.010	0.0034	0.063
B	0.0054	0.018	0.09	0.014	0.007	0.0032	0.10
C	0.0022	0.014	0.09	0.012	0.007	0.0072	0.069

Table 4 Fishscaling tendency of steels

Steel	SRT (°C)	D.P.* in enameling furnace (°C)		
		20	30	40
A	1 250	2/3	3/3	3/3
	1 150	2/3	3/3	3/3
B	1 250	0/3	0/3	0/3
	1 150	0/3	1/3	2/3
C	1 250	0/3	0/3	0/3
	1 150	0/3	0/3	0/3

* D.P.: Dewpoint of atmosphere

Steel	A (Low C, low N)	B (High C, low N)	C (Low C, high N)
Area fraction	0.118%	0.129%	0.174%
Mean diameter	1.07 μm	1.10 μm	1.26 μm

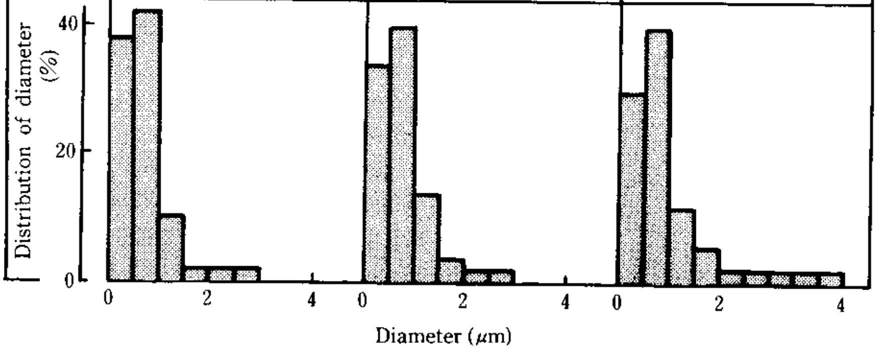


Fig. 7 Distribution of inclusion of steels

occurs even at a dew point of 20°C. Steel B with a higher C content than steel A shows different tendencies toward fishscaling depending on the SRT. That is to say, fishscale does not occur up to a dew point of 40°C whens the SRT is high, while it occurs at 30°C when the SRT is low. In steel C with a high N content, fishscale does not occur up to 40°C irrespective of the SRT. **Figure 7** shows results of observation of inclusions in these steel sheets with an optical microscope (× 800). Steel C has a larger area fraction and a larger mean diameter than other two steels. In addition, coarse inclusions 3 μm or more in diameter exist in steel C. From their shape, these coarse inclusions were judged to be TiN. The superior fishscale resistance of steel C compared to steels A and B is attributable to the existence of coarse TiN.

Steel B reheated to high SRT's and steel C can be put into practical use because porcelain enamel firing is scarcely conducted at high dew points above 30°C. It is advantageous, however, to use steel C that provides excellent fishscale resistance regardless of hot rolling conditions.

4.3 Effect of Ti Content of Steel on Porcelain Enamel Adhesion

Porcelain enamel adhesion varies greatly depending on conditions of porcelain enameling pretreatment such as pickling and Ni flashing. Pretreatment is an indispensable process for obtaining good adhesion especially when direct-on enameling is conducted using cover coating slip such as Ti opaque white slip. Enamel adhesion was examined on two types of steel sheets with different Ti contents by varying the pickling and Ni flashing time. The pickling bath used is a 10%H_2SO_4 solution (75°C) and the Ni flashing bath is a 2%$NiSO_4$ solution (65°C, pH = 3.0). The Ti opaque white slip made by Nippon Ferro Co., Ltd. (1553B, standard firing temperature: 820°C) was used. Adhesion was evaluated by conducting the P.E.I. (Porcelain Enamel Institute) adhesion test specified in ASTM C313-59. **Figure 8** shows results of a comparison of enamel adhesion between Ti-bearing steels and a decarburized capped steel (Kawasaki Steel specification: KTS-M). The steel containing 0.11%Ti shows deteriorated adhesion not only when the pickling iron loss is small, but also when it is large. Therefore, the range of pretreatment conditions for obtaining good enamel adhesion is narrow and the enamel adhesion of this steel is unstable compared with that of the decarburized capped steel. However, when the Ti content is decreased to 0.06%, good enamel adhesion is obtained in almost the same range of pretreatment conditions as with the decarburized capped steel.

Steel sheets were pickled with sulfuric acid and smut was removed by washing. As-pickled steel sheet surfaces and those after washing were observed under a scanning electron microscope. Results of this observation are shown in **Photo 2**. It is found that film-like smut is formed on the steel sheet containing a large amount of Ti. This smut was identified by X-ray diffraction as $FeSO_4 \cdot nH_2O$ in the Ti-bearing steel as well as in the decarburized capped steel. **Figure 9** shows changes in the smut weight as a function of pickling weight loss. The smut weight varies depending on the Ti content. The smut weight of the steel of 0.06%Ti is the same as that of the capped steel, while the smut weight of the steel of 0.11%Ti is heavier. **Figure 10** shows a comparison of porcelain enamel adhesion between a case where the steel sheet was enameled in the as-pickled condition and a case where it was enameled after the removal of

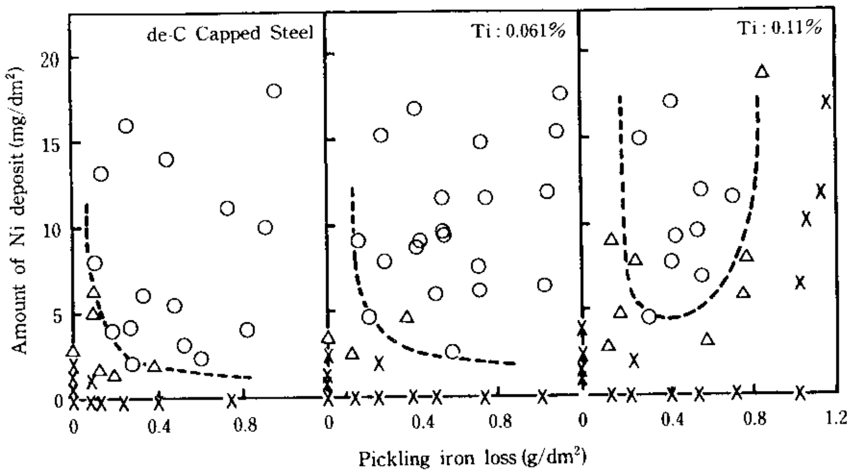
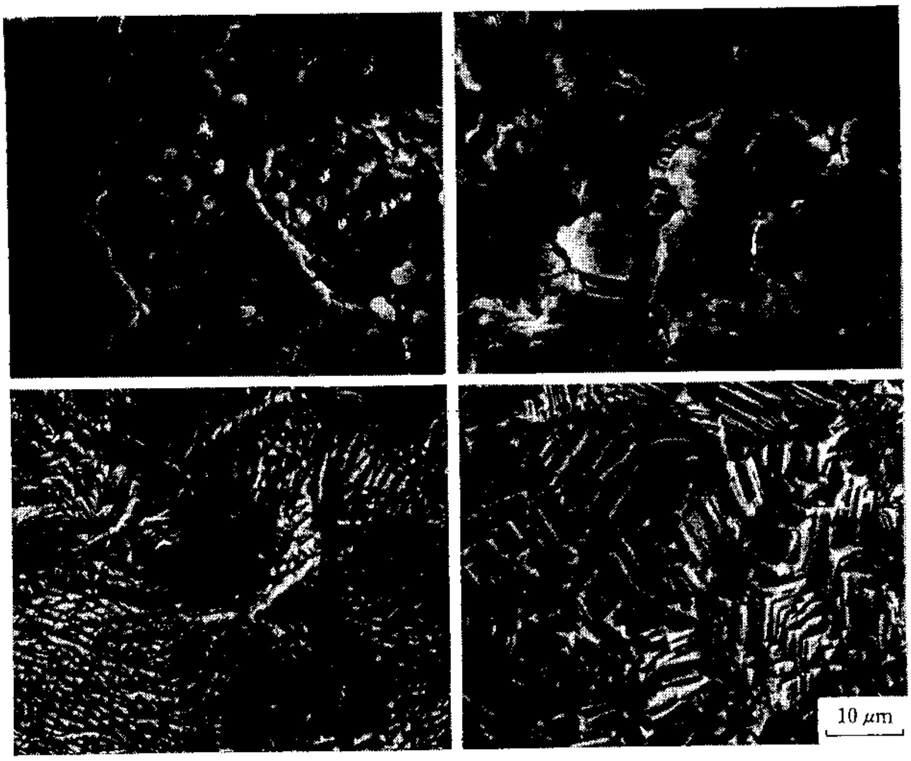


Fig. 8 Enamel adhesion of steels in direct-on enameling



(a) KTS-M (b) Ti-bearing steel

(Upper: as pickled Lower: after washing)

Photo 2 Surface image of steel sheet by SEM

smut by washing. Ni flashing was performed for 5 min as pretreatment. In the steel of 0.11%Ti, good enamel adhesion is obtained in the as-pickled condition only in a narrow range of pickling weight loss. In the steel of 0.06%Ti and of the decarburized capped steel, however, good enamel adhesion is obtained in wide ranges of pickling weight loss even if washing is not conducted. When smut is removed by washing, the enamel adhe-

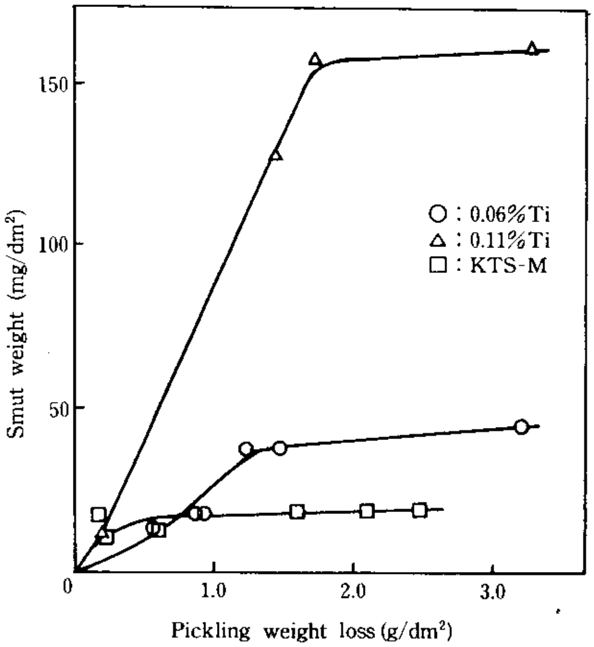


Fig. 9 Influence of pickling on smut deposition

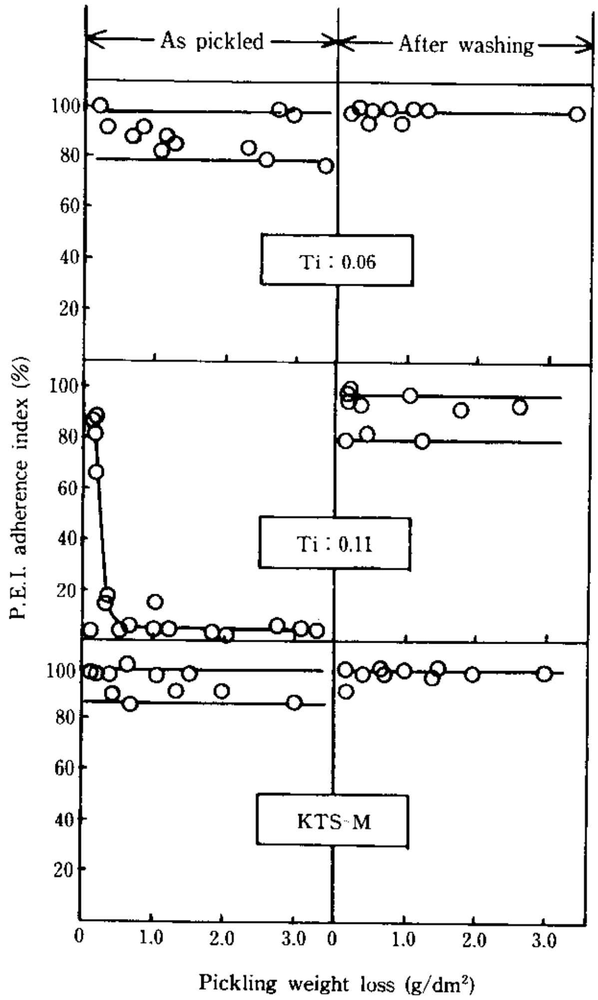


Fig. 10 Influence of smut on enamel adhesion

sion of the steel of 0.06%Ti and decarburized capped steel becomes excellent. In the steel of 0.11%Ti also, the enamel adhesion at large pickling weight losses shows a substantial improvement although there are variations.

It was found from the above-mentioned results that a large amount of smut is generated at high Ti contents of steel, thereby deteriorating enamel adhesion, while the same enamel adhesion as with decarburized capped steels can be obtained by reducing the Ti content to 0.06% or less.

4.4 Warping Characteristics

Steel sheets warp upon enamel firing when steel undergoes transformation or creep due to the thermal history of firing and when thermal strains are generated because of the different thermal expansion coefficients of steel sheet and porcelain enamel layer. To know the tendency toward deformation of steel sheets, therefore, the sagging test specified in ASTM-C22 was conducted at various temperatures on Ti-bearing steel sheets and decarburized capped steel sheets. **Figure 11** shows results of this test. The Ti-bearing steel does not show large sags up to high temperatures. This is because the carbon and nitrogen in steel are completely fixed as TiC and TiN, as a result of which the transformation point rises. Thermal expansion was measured after the heating at a heating rate of 10°C/s using Formaster to know the Ac_3 transformation point of these two types of steel sheet. The Ac_3 transformation point of the decarburized capped steel was 916°C and that of the Ti-bearing steel was 930°C. To examine the warping resulting from the difference in the thermal expansion coefficient between the steel sheet and enamel, slip was applied with various thicknesses to one side of each specimen of the same type as the ASTM-C22 specimen. Each specimen was subjected to 0 to 20% strains beforehand. When the enamel was fired while steel sheet was vertically suspended along its long side, the amount of warping was measured by the same method as specified for the sagging test. The L type ground coat slip made by Nippon Ferro Co., Ltd. (thermal expansion coefficient: 11×10^{-6}) was used. As shown in **Fig. 12**, the amount of warping is inversely proportional to the steel thickness and the yield stress (*YS*) of the steel sheet after firing, and is proportional to the porcelain enamel thickness. To reduce warping, therefore, it is necessary to reduce the difference in the porcelain enamel film thickness between the top and bottom surfaces and use steel sheets of appropriate thickness. It is also desirable that the decrease of *YS* of the steel sheet due to enamel

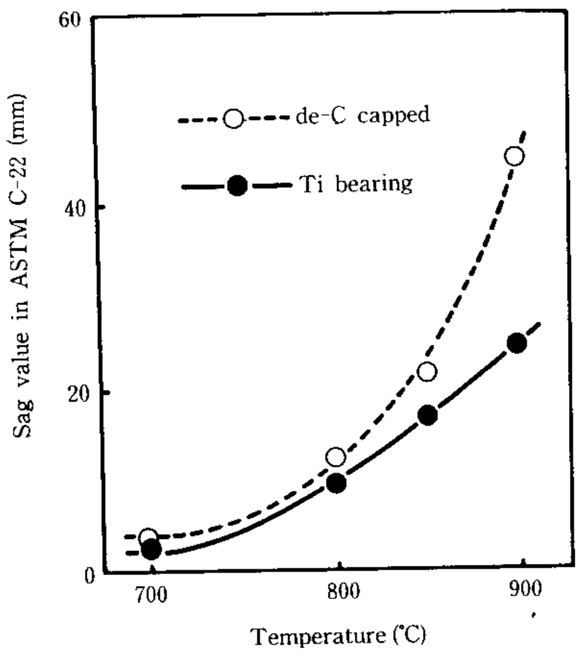


Fig. 11 Comparison of Sag value between de-C capped steel and Ti bearing steel

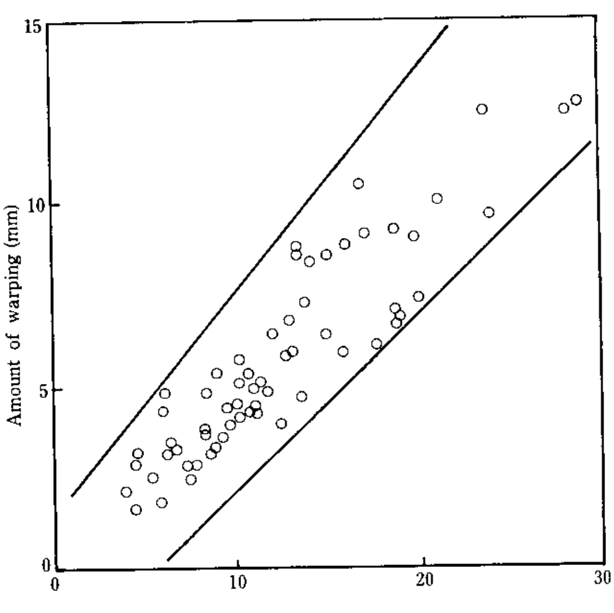


Fig. 12 Influence of enamel thickness, sheet thickness ane *YS* of steel on warping

firing is as small as possible. The same thermal history as enamel firing (820°C for 3 min) was given after the giving of various prestrains, and changes in the yield stress were examined. **Figure 13** shows results of this examination. The yield stress in the Ti-bearing steel does not decrease up to higher prestrains than in the decarburized capped steel. This means that the critical amount

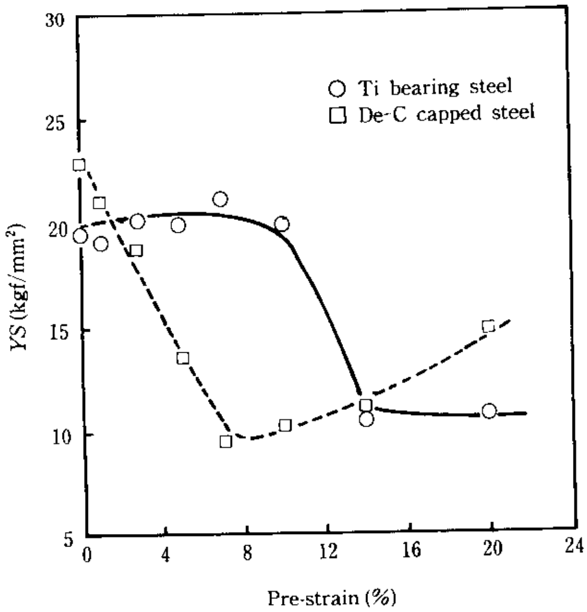


Fig. 13 Influence of pre-strain before firing on *YS* of steel sheets

of prestrain that causes grain growth is large. It can be said, therefore, that the Ti-bearing steel is superior to the decarburized capped steel in the deformation of steel sheets and the warping resulting from the difference in the thermal expansion coefficient between the steel sheet and enamel.

5 Conclusions

The mechanical properties and porcelain enameling properties of extra-low-carbon Ti-bearing steel sheets were investigated, and conditions for manufacturing enameling steel sheets with excellent press formability were explained.

Although fishscale is suppressed by the TiC and TiN in steel, the press formability of steel sheets deteriorates with increasing TiC. Therefore, it is beneficial to utilize TiN. When TiN is utilized, changes in the mechanical properties and fishscale resistance attributable to hot rolling conditions are small and it is possible to obtain steel sheets with $El > 50\%$ and $\bar{r} > 1.8$ even at low slab-reheating temperatures and low finisher delivery temperatures.

Ti contents of 0.06% or less are desirable because porcelain enamel adhesion decreases with increasing Ti contents.

However, a notable secondary working embrittlement occurs when steels with Ti contents of about 0.06% are box-annealed. For this reason, it is necessary to adopt continuous annealing at high cooling rates.

Ti-bearing steel sheets thus produced are superior to decarburized capped steel sheets so far widely used for porcelain enameling in press formability and warping characteristics. They provide stable porcelain enamel adhesion and fishscale resistance.

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