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Effect of Nitrogen and Other Alloying Elements on the Low-Temperature Brittleness of Steel. III
Nitrogen Fix and Nitrogen in Quench-Tempered Steel*

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Synopsis

The following have been made clear by examining the effect on the low-temperature brittleness of steel of aluminium and titanium as the elements fixing nitrogen. (1) The effect of nitrogen decreases with the addition of aluminium and titanium; (2) in the steel containing 0.32% of carbon, AlN lowers a little the pulse value of steel, but has little effect on the transition temperature; (3) unlike the case of adding aluminium, the addition of titanium slightly raises the maximum pulse value and tends to raise the transition temperature; (4) when the effect of nitrogen on the low-temperature brittleness of the steel containing phosphorus appears relatively large, the addition of titanium clearly improves the low-temperature brittleness of the steel; (5) the effect of nitrogen can be observed even in the steel quench-tempered, the effect of nitrogen similar to the case of air-cooling appears on slow-cooling after tempering and the aging phenomenon appears on rapid-cooling after tempering; (6) in both cases, the transition temperature rises proportionally to the amount of nitrogen content.

I. Introduction

It has been made clear in the previous study[1] that the effect of nitrogen on the low-temperature brittleness decreases when nitrogen is fixed by aluminium added. In the present study comparisons were made between the effects of aluminium, titanium and nitrogen on soft steel subjected to quench-tempering treatment cooled from 950°C as described in Report I[2], that is, the effect in which the low-temperature brittleness became greater and the transition temperature rose with the increase of nitrogen content. In these experiments, the impact values were determined in the temperature range of −196° to 200°C by U-notch Charpy impact test, and the transition temperatures were determined. On the assumption that the temperature — impact energy curve would be smooth, from the maximum impact value, the impact value at 0°C and the arithmetical mean of the maximum and the minimum impact values.

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* The **1186th** report of the Research Institute for Iron, Steel and Other Metals.

** Technical Development Department, Fuji Iron and Steel Co., Ltd.

(1) T. Ishizaki and Y. Imai, Sci. Rep. RITU, **A 17** (1965), 70.

(2) T. Ishizaki and Y. Imai, Sci. Rep. RITU, **A 17** (1965), 61.

II. Experimental result

1. Effect of AlN in carbon steel

It has been made clear in the previous study that if nitrogen is fixed by aluminium, it makes the impact value higher and the transition temperature lower. The aluminium added will combined with nitrogen, aluminium nitride being formed, but a part of aluminium dissolves in ferrite, and it is also natural that Al_2O_3 is partially formed. To see easily the effect of AlN, the experiment was performed on the assumption that almost all nitrogen exists in the form of AlN. The specimens were prepared by adding nitrogen to steels containing 0.32% and 0.43% of carbon, respectively. Other specimens were prepared by adding nitrogen to steels containing 0.35% of carbon and 0.1% of phosphorus (Fig. 1). It will be seen that aluminium fixes the effect of a large amount of nitrogen as well as a small nitrogen content (0.006 % N) shown in Figs. 10 and 20 in the previous report.

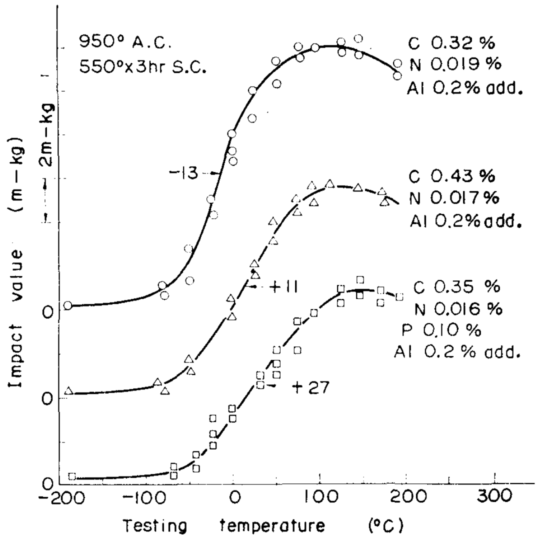


Fig. 1. Effect of temperature on impact value of steels, contained large amount of N.

In Figs. 2 and 3 are shown the effect of nitrogen content (A1N) on the impact value and the transition temperature: the maximum impact value and the impact value at 0°C are both small in the specimens containing much nitrogen, but any change is hardly observable in the transition temperature. In this case the temperature at which the impact value is 5 mkg (an approximate average of the impact values at the transition temperature) shown for reference, did not show any change.

It seems that the effect of AlN of lowering the impact value of steels containing more than 0.32% of carbon is slight, compared with that of low-carbon steels, hardly affecting the transition temperature.

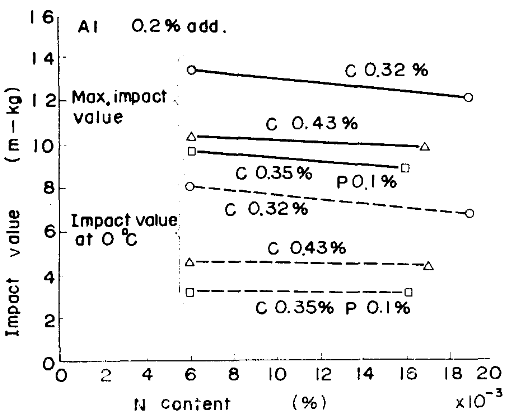


Fig. 2.

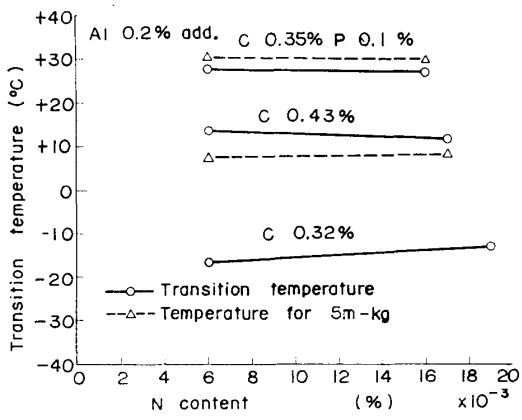


Fig. 3.

Fig. 2. Effect of N content on impact value of steels, added Al.
Fig. 3. Effect ot N content on transition temperature of steels, added Al.

2. The effect of nitrogen fixed by titanium in carbon steel

Similar to the case where nitrogen is fixed by aluminium, it is expected that the addition of titanium may reduce the effect of nitrogen in ferrite. Titanium thus added forms compounds TiN (Ti-C-N, Ti-C-N-O), a part of which dissolves in ferrite as metallic titanium, as in the case of aluminium; dissimilar to the case of aluminium, titanium forms TiC when carbon is present. It has been made clear that when titanium is added, it will combine first with oxygen and then with nitrogen and finally with carbon, sulphur, etc.(3) The specimens used were steels containing 0.32% of carbon and 0.006% of nitrogen (Fig. 4), to which 0.075% of titanium was added, and those containing 0.36% of carbon, 0.1% of phosphorus and 0.006% of nitrogen (Fig. 6) to which 0.30% of titanium was added.

In Fig. 5 are shown the results obtained from the steels containing no phosphorus, in which it is seen that the maximum impact values rise and the impact values at 0°C slightly fall with the addition of titanium, and that the transition temperature shows the highest value when 0.075% of titanium is added, beyond which it tends to lower. Although the change in the maximum impact values is similar to that in the steels containing aluminium, the change in the transition temperature (the change in the impact values at 0°C can be explained in the way similar to this case, as they are under the effect of the transition temperature) is dissimilar to that in the steels containing aluminium. Because the specimens used in the present experiments contain less nitrogen, the effect of raising the transition temperature by TiN promotes the nitrogen fixing effect by titanium. It seems probable that if the experiments are performed with specimens containing much nitrogen, the effect of nitrogen fixing will be so great that the transition temperature will fall.

The results obtained from the specimens containing phosphorus are shown in Fig. 7, in which it is clearly observable that the addition of titanium reduces the

(3) T. Saito, Tetsu-to-Hagané, **39** (1953), 29.

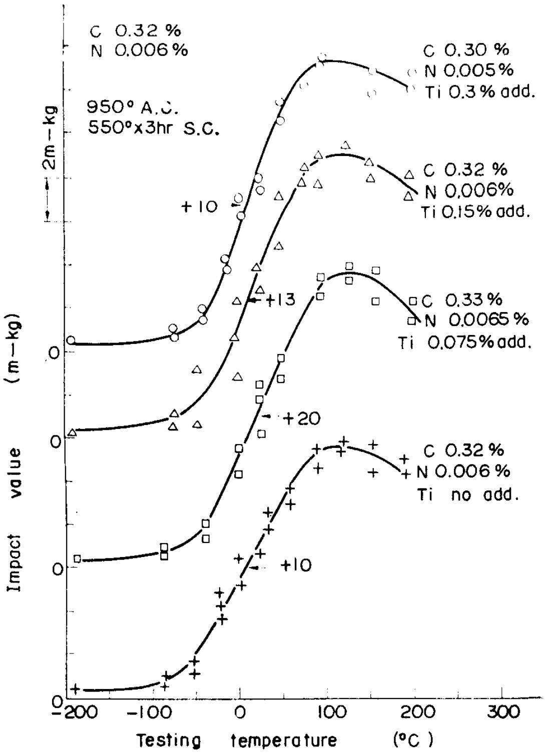


Fig. 4.

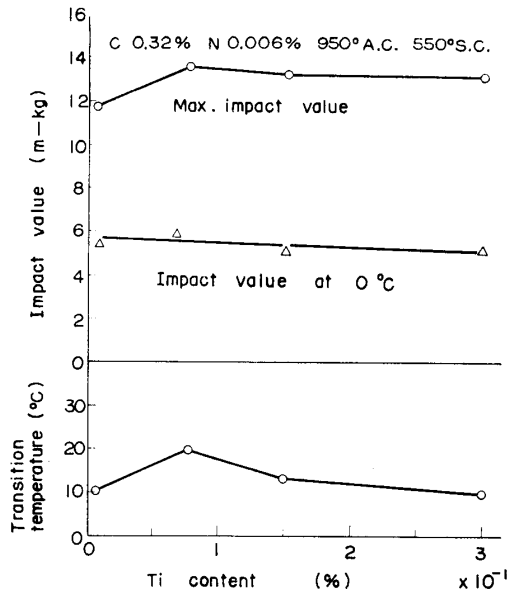


Fig. 5.

Fig. 4. Effect of temperature on impact value of steels, added Ti.
Fig. 5. Effect of Ti content on impact value and transition temperature.

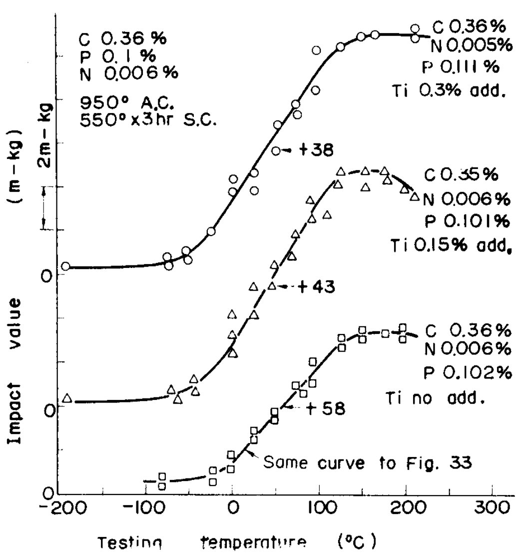


Fig. 6.

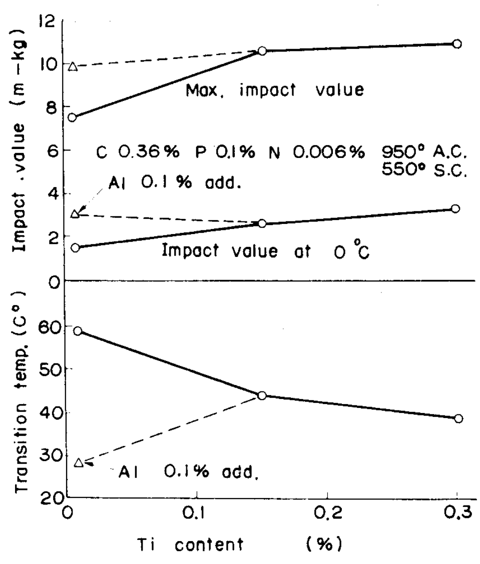


Fig. 7.

Fig. 6. Effect of temperature on impact value of steels, contained P and added Ti.
Fig. 7. Effect of Ti content on impact value and transition temperature.

effect of nitrogen, that is, that the effect of nitrogen is much greater, and the fixing effect of nitrogen is clearly observed. In this case the effect of raising the transition temperature caused by titanium should be present. This can be seen by comparing it with the values of the specimens (△ mark), to which only aluminium is added, shown in the figure. When nitrogen is fixed by aluminium, titanium, etc., the fall in the impact value and the rise in the transition temperature caused by nitrogen can be improved. Attention is called here to the fact that AlN and TiN will reduce the maximum impact value and that the addition of titanium will raise the transition temperature.

3. The effect of nitrogen in quench-tempered steel

As described in Report I, the effect of nitrogen was greater in the normalized steels containing less carbon, and so experiments were performed with the specimens prepared by heating the steel containing 0.02 to 0.03% of carbon, 0.2 to 0.3% of manganese and 0.15 to 0.21% of silicon at 950°C for 30 minutes, quenched in water, tempered at 620°C for 1 hour, cooled in water or slowly cooled at the rate of 1 degree per minute, and aged at room temperature for a sufficient time (about 30 days), in order to obtain the results to be compared with those mentioned in the above. By making use of the specimens left over, they were reheated at 620°C and cooled in water for impact test, to obtain the values when they were not aged. Approximate transition temperature were determined, but they were inaccurate

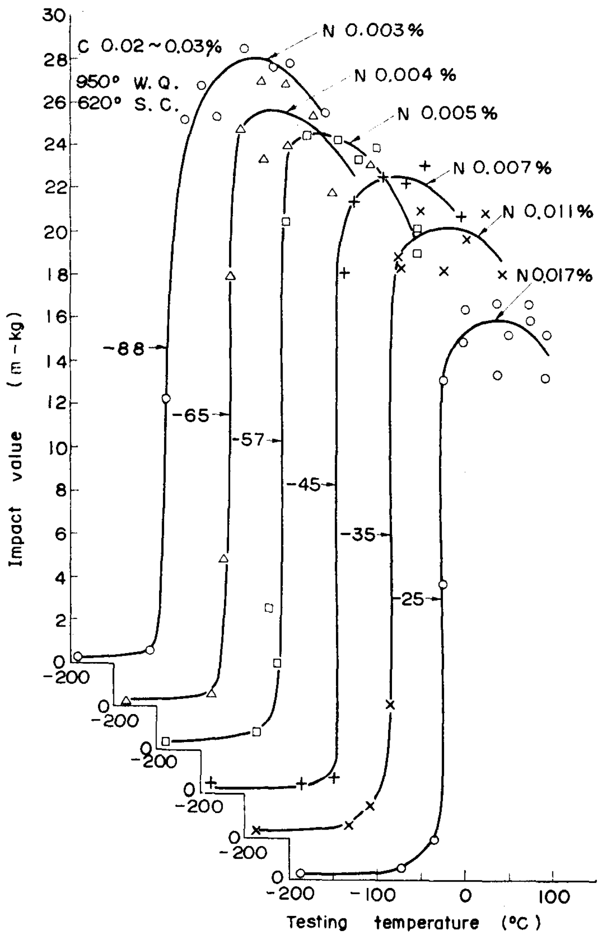


Fig. 8. Effect of temperature on impact value of 0.03% C steel, slow cooled.

because only a few specimens could be used.

Fig. 8 shows the results obtained from the specimens slowly cooled after tempering; Fig. 9 those from the specimens cooled in water after tempering and

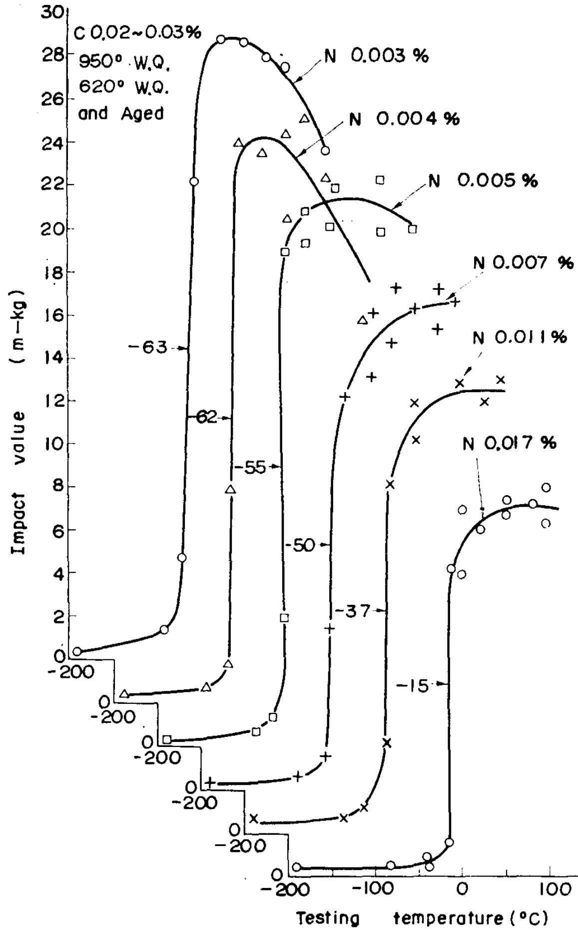
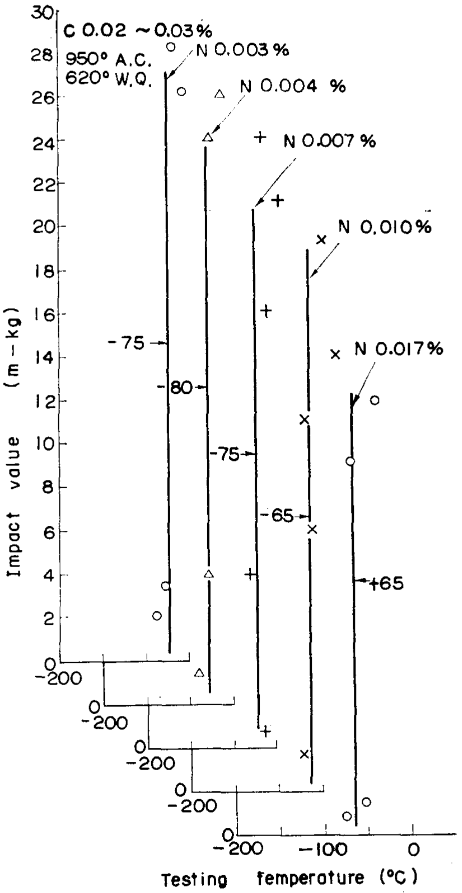


Fig. 9. Effect of temperature on impact value of 0.03% C steel, water-quenched and aged.

aged; Fig. 10 those from the specimens cooled in water after tempering and subjected to the impact test as soon as possible to remove the effect of aging; Fig. 11 those from the specimens in which nitrogen was fixed first by adding 0.014 to 0.015 % of nitrogen and 0.1% of aluminium and then 0.2% of aluminium. Fig. 12 shows the relation between the maximum value and the nitrogen content, from which it is seen that although the nitrogen content is larger in the specimen to which aluminium is added, the greater part of it is fixed as AlN, and that the nitrogen directly affecting the low-temperature brittleness is small, as described in Report II. Consequently, they are shown in the left side of the figure, that is, they are shown in the place of specimens containing low-nitrogen content. The maximum impact values became lower slowly with the increase of nitrogen in the slowly cooled specimens, but rapidly in the quench-aged specimens. The difference in the impact values between the slow-cooled and the quench-aged specimen becomes large with the



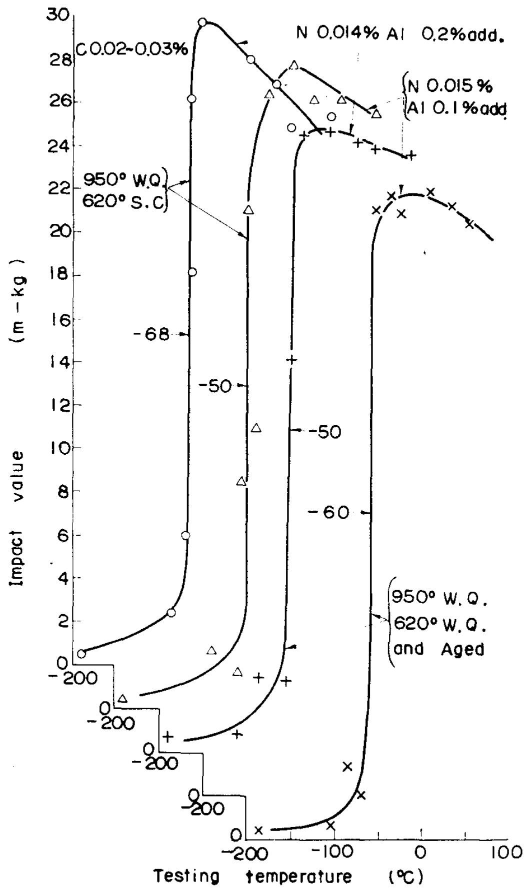
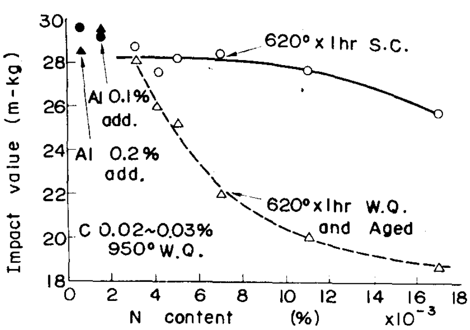


Fig. 10.

Fig. 11.

Fig. 10. Effect of temperature on impact value of 0.03% C steel, water-quenched.
Fig. 11. Effect of temperature on impact value of Al killed 0.03% C steel.



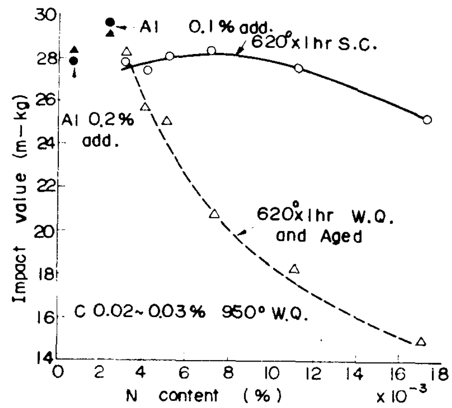


Fig. 12.

Fig. 13.

Fig. 12. Effect of N content on max. impact value of 0.03% C steel.
Fig. 13. Effect of N content on impact value at 0°C of 0.03% C steel.

increase of nitrogen content. The impact values of the specimens in which nitrogen is fixed by aluminium added are high in all cases.

Fig. 13 shows the relation between the impact values at 0°C and the nitrogen content; the results are so similar to those of the maximum impact values that no explanation will be necessary. Fig. 14 shows the relation between the transition temperature and the nitrogen content; in the slow-cooled specimens, the transition temperature rapidly rises (about 10°/0.001% N) until about 0.008% N,

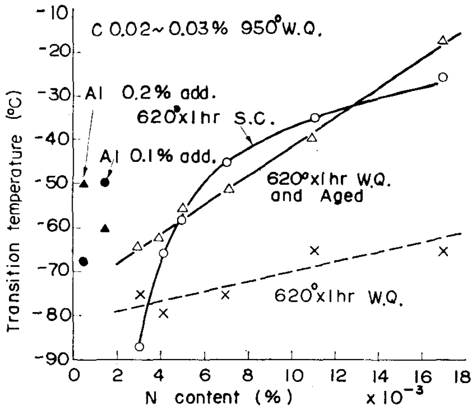


Fig. 14. Effect of N content on transition temperature of 0.03% C steel.

beyond which it rises slowly (about 1.5°/0.001% N). In water-quenched and quench-aged specimens, the transition temperature in the water-quenched specimens is lower than that in the quench-aged specimens, the difference becoming greater with the increase of nitrogen content. In other words, in the water-quenched specimens, the transition temperature rises with the increase in the solubility of nitrogen in ferrite (about 1°/0.001 % N) and the transition temperature rises proportionally to nitrogen content when the specimens are aged at room temperature (about 7°/0.001% N). It can be said, therefore, that it is nitrogen that causes the rise of transition temperature by aging. When nitrogen is dissolved in ferrite, the rise in the transition temperature due to a strain by solution is slight, and nitrogen in the precipitated or in the segregated state will show a remarkable resistance against slip deformation and the transition temperature will much rise as seen in the quench-aged specimen. Similar to the case shown in Fig. 13 in Report I, it is because the nitrogen precipitated at the temperature below 300°C severely raises the transition temperature that a bend is shown in the slow-cooled specimen containing about 0.008% of nitrogen. The soluble nitrogen in the specimen to which aluminium is added ought be small, but it shows a relatively high transition temperature; in the case of 0.1% of aluminium being added, the transition temperature is higher in the slow-cooled specimen than in the quench-aged specimen; on the contrary, in the case of 0.2% of aluminium being added, the transition temperature is higher in the quench-aged specimen than in the slow-cooled specimen. A detailed discussion on such a phenomenon will be given in another paper.

Suffice it to say here that in the case of aluminium being added, the transition temperature falls with the progress in the process of fixing nitrogen in the slow-cooled specimen, while in the quench-aged specimen, with the appearance of the effect of carbon dissolved in ferrite, the aged amount will be larger with the progress in the process of fixing nitrogen. An inference can be made from the results of studies on the blue brittleness(4), quench-aging(5) and strain-aging(6) on these phenomena.

Summary

By studying the effect of AlN in the steel containing 0.32% of carbon the following have been made clear.

(1) AlN has little effect on the transition temperature, although it lowers slightly the maximum impact values.

(2) TiN lowers the maximum impact value and raises the transition temperature.

(3) The effect of nitrogen on the low-temperature brittleness of the steel is clearly observable even if it is subjected to quench-tempering treatment, and the low-temperature brittleness becomes sharper with the increase of nitrogen content.

(4) Y. Imai and T. Ishizaki, Sci. Rep. RITU, **A 4** (1952), 553.
(5) Y. Imai and T. Ishizaki, J. Japan Inst. Metals, **17** (1953), 318.
(6) Y. Iami and T. Ishizaki, J. Japan Inst. Metals, **18** (1954), 524; **19** (1955), 194.