Hardness and Structure of Ausformed Fe-Ni and Fe-Ni-C Alloys*

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The effects of ausforming on the hardness and structure of Fe-Ni and Fe-Ni-C alloys were examined. The results of microhardness tests showed that some amount of the carbon content might be essential for the strengthening of steel by ausforming. Optical micrographs showed that a martensite plate in the ausformed specimen was finer than that in the unausformed, and was bent and kinked. Transmission electron micrographs and electron diffraction patterns of ausformed Fe-Ni-C alloy showed that : (1) There were strong stresses within and around the martensite plate. (2) The twin faults in the martensite crystal were also recognized. (3) A cloudy distribution of dislocations was observed in the martensite plate, and the carbon atoms might migrate on these dislocations. The ausformed specimen was hardened mainly by precipitation and/or dispersion of carbides. (4) Severely kinked martensite plates were frequently observed. (5) The lattice orientation relationship between austenite and martensite in the ausformed Fe-Ni-C alloy approximately obeyed the usual law in the same way as in the unausformed.

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I. Introduction

When a steel is deformed at a metastable austenitic state, transformed into martensite and then tempered, the steel is much more strengthened than an ordinarily quenched and tempered steel. Since such a phenomenon was discovered by Harvey⁽¹⁾ in 1951, it has been investigated and developed by several researchers⁽²⁾⁻⁽¹⁶⁾, and such a thermal-mechanical treatment

is called "Ausforming".

But, the reason for the strengthening of steel by ausforming has not been fully clarified yet, and many

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problems remain to require further precise observations and examinations. It is expected to find some evidences by transmission electron microscopy as already suggested by Justusson et al⁽¹⁴⁾ and Radcliffe et al⁽¹⁷⁾. This paper reports on the results of some observations of the ausformed Fe-Ni and Fe-Ni-C martensites mainly by transmission electron microscopy.

II. Experimental Procedure

Specimens were prepared from electrolytic iron and electrolytic nickel by melting and casting in vacuum. They were hot forged and then cold rolled into 0.65 mm-thick plates. Some of the plates were used as Fe-Ni specimens and others were carburized at 950°C for

6 hr and homogenized in vacuum at 900°C for 24 hr to make Fe-Ni-C specimens. The chemical compositions of the Fe-Ni and Fe-Ni-C specimens are shown in Table 1.

Table 1 Chemical compositions and reduction by ausforming of Fe-Ni and Fe-Ni-C alloys.

Alloy	Ni %	C %	Fe %	% ausformed
Fe-Ni	29.7	0.006	bal.	0 %, 40 %
Fe-Ni-C	29.7	0.420	bal.	0 %, 24 %, 30 %

The specimens were austenitized at 900°C quenched in an oil bath at 200°C. After being held in the bath for 2 min, they were worked by rolling and air-cooled to room temperature. In the investigation by optical microscopy, no martensite was found in the Fe-Ni-C specimens, but an amount of martensite was observed in the Fe-Ni specimens at room temperature. These specimens were then dipped in liquid nitrogen for 30 min. By this treatment, the transformation into martensite was almost completed in the Fe-Ni specimens but about half in the Fe-Ni-C specimens. Specimens which had not been subjected to rolling were also used for reference, designating them 0%-ausformed specimens.

The microhardness of these specimens was measured and the structures were observed by optical microscopy and transmission electron microscopy. Thin foils for the transmission electron microscopy were prepared by Bollmann's method in an electrolyte composed of phosphoric acid and chromic acid at 80°C. The electron microscope used is a Hitachi HU-11, operated at 100 KV.

III. Experimental Results and Considerations

1. Hardness

Fig. 1 shows the results of hardness measurements. In the case of 500g-loading, the measurement was made irrespectively of the microstructure, while in the case of 50g-loading it was made for martensite and austenite separately.

In 500g-loading, the Fe-Ni specimen was scarcely hardened, but the Fe-Ni-C specimen was markedly hardened by ausforming. The hardness of austenite in the Fe-Ni specimen was measured before deep-cooling in liquid nitrogen because after deep-

Fig. 1 Hardness of martensite and austenite in ausformed Fe-Ni and Fe-Ni-C alloys measured by 50g-loading and hardness of the same alloys for 500g-loading. Each point-10 readings. [] represent spread.

cooling the austenite was markedly reduced in quantity, but in the Fe-Ni-C specimen it was measured for austenite retained after deep-cooling. In the Fe-Ni specimen the austenite was hardened by straining, but the extent was smaller than that in the Fe-Ni-C specimen. The martensite in the Fe-Ni specimen was scarcely hardened by ausforming. In the Fe-Ni-C specimen, however, not only the austenite but also the martensite was markedly hardened by ausforming. Thus, the effect of ausforming was clearly found in the Fe-Ni-C alloy, but not in the Fe-Ni alloy. It may, therefore, be considered that some amount of the carbon content is essential for the strengthening of steel by ausforming.

But, Justusson et al⁽¹⁴⁾ have shown that the increment of yield strength of steel by ausforming does not depend on the carbon content. However, their experiments were carried out for a series of steel containing more than 0.3% carbon. The results can be explained that an excessive amount of carbon is not effective on the strengthening of steel by ausforming, although some amount of carbon is required to increase the strength by segregation to the dislocations and saturating them. A small amount of the carbon content may be sufficient to saturate the dislocations, depending upon the dislocation density which is high in the case of the ausformed specimen as shall be described later.

(17) S.V. Radcliffe and E.B. Kula: "Deformation, Transformation and Strength", presented at the Conference on Fundamentals of Deformation Processing, Sagamore, N.Y., Aug. 1962.

2. Optical microscopic structure

Photo. 1 shows optical micrographs of the Fe-Ni-C specimens in the 0% - and 30% -ausformed states after deep-cooling. Both of them have a lightening or zigzag shaped martensite. The structure of the 30% -ausfor-

ted by Nishiyama and Shimizu⁽¹⁸⁾⁽¹⁹⁾ and by Kelly and Nutting⁽²⁰⁾⁽²¹⁾ to two kinds of crystals which had a twin relation to each other. The features of the structure in Photo. 2 are such that the fine parallel bands run through the whole martensite plate, and

(A)

(B)

Photo. 1 Structures of (A) 0% - and (B) 30% -ausformed Fe-Ni-C alloys taken by an optical microscope. $\times 200$, Etchant: 1% Nital.

Photo. 2 Transmission electron microstructure of a 0% -ausformed Fe-Ni-C alloy.

med specimen is, however, finer than that of the 0% -ausformed, and the martensite plates are small, and warped, bent and kinked, while those in the 0% -ausformed are large and straight.

The above facts are in agreement with those reported by several researchers.^{(4)~(7)(9)(11)(14)} Such a structure in the ausformed specimen may be produced due to the interaction of the martensite plate in growth with slip bands and deformation bands existing in the deformed austenite⁽¹¹⁾⁽¹⁴⁾.

3. Structures observed by transmission electron microscopy

Photo. 2 shows a transmission electron micrograph of martensite and retained austenite in the 0% -ausformed Fe-Ni-C specimen. There are fine parallel bands having an interval of 100-400Å in the martensite. Such a structure of martensite has been attrib-

uted to the shape of bands at the ends (interface between martensite and austenite) indicates them to be plate-like. Furthermore, there are some lines and dark dotted images between the bands. They are explained as contour fringes and fine precipitates of carbide on the twin faults, respectively. The micrograph is very clear and dislocations in the martensite are hardly observed. The structure of austenite is smooth, and there are a few smooth contour fringes and dislocations.

The diffraction pattern of selected area (a) in the martensite in Photo. 2 is shown in Photo. 3. This pattern indicates that there are martensite crystals of

(18) Z. Nishiyama, and K. Shimizu: *Acta Met.*, **7** (1959), 432.

(19) K. Shimizu: *J. Phys. Soc. Japan*, **17** (1962), 508.

(20) P. M. Kelly and J. Nutting: *J. Iron Steel Inst.*, **197** (1961), 199.

(21) P. M. Kelly and J. Nutting: *Proc. Roy. Soc.*, **259** (1960), 45.

two kinds of orientations which have a twin relation to each other with respect to the $(2\bar{1}1)$ plane, and the fine bands in the structure of Photo. 2 are the trace of the twin faults. Photo. 4 shows the diffraction pattern of selected area (b) in austenite in Photo. 2.

Photo. 3 Electron diffraction pattern of area (a) in Photo. 2.

Photo. 4 Electron diffraction pattern of area (b) in Photo. 2.

It has been recognized that the habit plane of martensite is $\{259\}_r$ and that the lattice orientation relationship between austenite and martensite is approximately $(1\bar{1}1)_a // (10\bar{1})_r$, $[\bar{1}0\bar{1}]_a // [\bar{1}1\bar{1}]_r$, which agree with those reported so far.

Photo. 5 shows an electron micrograph of the 30%-ausformed Fe-Ni-C specimen. In the martensite, there are several diffused and broad lamellar bands. These bands are extinction contour fringes.

Zackay et al.⁽¹¹⁾ have reported the morphology of martensite formed from deformed austenite, as observed by optical microscopy. According to them, slip bands, deformation bands and sub-boundaries in the deformed austenite are semipermeable barriers to the passage of the martensite plate. When the martensite plate grows passing through the slip bands or deformation bands, the growth process and the shape of

the plate are changed. It may therefore be expected that the martensite plates are bent and distorted at those barriers in the austenite, and frequently the growth no longer continues. Finally, high stresses of wide range are retained within and around the mar-

Photo. 5 Transmission electron microstructure of a 30%-ausformed Fe-Ni-C alloy showing the broad bands in martensite.

Photo. 6 Electron diffraction pattern of area (a) in Photo. 5.

tensite plate, and the plate becomes finer. When the thin foil for transmission electron microscopy is made from a bulk material having such retained stresses, it would be bent locally by the stresses. In such a case, many extinction contour fringes can be observed in the micrograph. The fringes in Photo. 5 are the typical examples.

Photo. 6 shows an electron diffraction pattern of the ausformed martensite at the selected area of Photo. 5. The twinned pattern is also observed. Each of the diffraction spots located on the lower-left and the upper-right sides in Photo. 6 consist of two or three separate spots. The direction of separation is approximately perpendicular to the direction of the broad bands in Photo. 5. The separation is probably caused by the fact that the thin foil is bent wavy around the direction of the lamellar broad bands and

hence the lattice is severely distorted.

The most important peculiarity of martensite in the ausformed specimen is that the image of the martensite is not clear as observed in Photo. 5. Such an image shows the existence of a cloudy distribution of dislocations at which extremely fine particles of carbide may precipitate. The high dislocation density in the ausformed martensite is made more clearly obser-

ties of ausformed martensite.

4. Lattice orientation relationship between austenite and martensite in the ausformed Fe-Ni-C alloy

Photo. 8 shows a structure of the 30%-ausformed Fe-Ni-C alloy. The electron diffraction patterns of selected area (a) (martensite) and (b) (austenite) in

Photo. 7 Transmission electron microstructure of a 30%-ausformed Fe-Ni-C alloy showing the kinking of martensite plate.

Photo. 8 Transmission electron microstructure of a 30%-ausformed Fe-Ni-C alloy.

vable by tempering ⁽²²⁾.

The twin faults are also observed in the ausformed martensite plate (b) in Photo. 5 in the same manner as in the unausformed martensite. But, usually, it is difficult to observe the twin faults in the ausformed martensite because of hindrances of several images of dislocations, fringes and others.

A dense distribution of dislocations is observed in the austenite in Photo. 5. The austenite is evidently strain-hardened.

Photo. 7 is another electron micrograph of the 30 %-ausformed Fe-Ni-C specimen. The martensite plate is kinked corresponding to the kinking seen in the optical micrograph. This is also one of the peculiari-

ties of ausformed martensite. Photo. 8 are shown in (A) and (B) of Photo.9, respectively. The lattice orientation relationship between austenite and martensite is approximately $(111)_\gamma // (011)_\alpha$ and $[\bar{1}\bar{1}0]_\gamma // [\bar{1}\bar{1}1]_\alpha$. Thus the usual relationship holds even in the ausformed state. Kula et al⁽²³⁾ have determined the preferred orientation of rolled austenite assuming that Nishiyama's relationship holds even in ausformed specimens. The results obtained from Photo. 9 prove their assumption to be correct.

5. Consideration on the strengthening by ausforming

According to Winchell et al⁽²⁴⁾, the martensite even

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(23) E. B. Kula and S. L. Lopata : Trans. AIME, 215 (1959), 980.

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in the unausformed specimen can be gradually age-hardened at room temperature due to segregation of carbon atoms mainly on the twin faults⁽²⁾. In the ausformed martensite, there are a wide range internal stress and a cloudy distribution of dislocations. Since

the dislocations are inherited from the deformed austenite or they are produced in the martensite to accommodate the stress during or after martensite formation. But, the former might be able to explain the strengthening of steel by ausforming.

Generally, alloys hardened by precipitation or dispersion of fine particles are reduced in ductility, namely, elongation and reduction of area. Lower ductility in the technological sense means a large possibility for the fracture of material. When numerous dislocations are piled up at a few obstacles in the material during the tensile test, a large stress concentration occurs and the material is easily fractured with the resulting decrease in ductility. On the contrary, when few dislocations are piled up at numerous obstacles which are densely distributed over the material, the stress is not so much concentrated and the material does not become so brittle even in the hardened state.

The ausformed steel has fairly good ductility.⁽¹¹⁾⁽¹⁴⁾⁽¹⁷⁾ For this reason, it may be expected that in the ausformed steel there are some factors such as the fineness of martensite plates which reduce the stress concentration, together with the factors for strengthening of steel as described above.

IV. Summary

The hardness and the structures of ausformed Fe-Ni and Fe-Ni-C alloys have been examined. The results are summarized as follows:

- (1) Some amount of the carbon content is essential for the strengthening of steel by ausforming.
- (2) The ausformed martensite is harder and finer than the unausformed, and is bent and kinked.
- (3) In the ausformed martensite, densely distributed dislocations at which carbon atoms segregate are observed.
- (4) A large internal stress of wide range are recognized in the ausformed martensite.
- (5) Usual lattice orientation relationship between austenite and martensite holds good even in the ausformed specimen.
- (6) The ausformed steel has a high strength due mainly to precipitation and/or dispersion of fine carbides on the dense dislocations, and has a fairly good ductility due to some factors such as the fineness of martensite plates which reduce stress concentration, together with the strengthening factors.

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Photo. 9 Electron diffraction patterns of area (a) and (b) in Photo. 8.

the nucleation of carbide is enhanced by the presence of dense dislocations, the martensite of the ausformed specimen becomes a pre-precipitated state or an age-hardened state more steadily, and the precipitated carbide particles are finer and more closely dispersed than those in the unausformed. Furthermore, the martensite plate becomes finer and is bent and kinked by ausforming. Therefore, the martensite in the ausformed specimen is harder than that in the unausformed.

As for the origin of dislocations in the ausformed martensite, difficulty is encountered to decide whether