Serrated Flow and Dynamic Precipitation in Elevated Temperature Tensile Deformation of Fe-Mn-Al-C Alloys

K. S. Chan[†], L. H. Chen and T. S. Lui

Department of Materials Science and Engineering, National Cheng-Kung University, Tainan, Taiwan 70101, R. O. China

To investigate the characteristics and effects of dynamic strain aging (DSA) and dynamic precipitation of austenitic Fe-Mn-Al-C alloys, a hot-rolled alloy (Fe-26Mn-9Al-0.9C, in mass%) and two cast alloys (Fe-35Mn-9Al-1C, Fe-32Mn-9Al-1C) were selected to perform tensile test at various temperatures from 298 to 1073 K. The strain rates were chosen in the range from 9.4×10^{-5} to $9.4 \times 10^{-3} \text{ s}^{-1}$.

As indicated from the appearance of serrated flow, DSA occurs in two temperature regimes. Judging from the apparent activation energy obtained at the onset temperatures of serration, carbon atmosphere drag plays a role in the serration and the temperature independent flow stress in the low temperature regime. In the high temperature regime, the serration cannot be solely accounted for by the diffusion of manganese or aluminum solute atoms, or both. During high temperature deformation, the precipitation of Fe_3AlC_x occurs on active slip planes and along grain boundaries, giving rise to a substantial strengthening effect as compared with that normally associated with DSA. Grain boundary precipitation also leads to grain boundary embrittlement at the temperatures higher than about 773 K. All the above features and effects are common in the three test alloys although the hot-rolled alloy contains about 4 vol% ferrite.

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I. Introduction

Since nickel and chromium are expensive strategic alloying elements, the substitution of conventional Ni-Cr stainless steels by austenitic Fe-Mn-Al-C alloys has become of interest⁽¹⁾. With a proper composition to form protective scale, austenitic Fe-Mn-Al-C alloys are good in corrosion and oxidation resistance⁽²⁾⁻⁽⁴⁾. The alloys are also high in strength and ductility at and below room temperature⁽⁵⁾⁻⁽⁹⁾. With some decrease in ductility, the strength of these alloys can be raised substantially by static aging at about 723 to 973 K⁽¹⁰⁾⁻⁽¹³⁾. The age-hardening effect is due to the formation of κ -phase Fe_3AlC_x precipitates⁽¹⁰⁾⁻⁽¹³⁾. To give some quantitative data, Hale *et al.*⁽¹¹⁾ have reported that the room temperature 0.2% off-set yield strength of a hot-rolled Fe-31Mn-8Al-0.97C (composition in mass%, hereafter) was increased from 450 to 850 MPa after aging at 823 K for 5 h. The elongation of the aged material was about 40% as compared to 70% of the unaged material.

High temperature mechanical property data are important if the alloys are to be hot-worked or used in high-temperature structural applications. To the authors' knowledge, these data are rare and incomplete. For example, the occurrence of dynamic strain aging (DSA), which is characterized by serrated flow, has only been men-

tioned without any detail⁽¹⁴⁾. Also, brittle fracture has been found in tensile deformation at the temperatures around the precipitation temperature for Fe_3AlC_x ⁽⁹⁾⁽¹⁵⁾. Again, no details of the microstructure evolution corresponding to this embrittlement have been mentioned.

This paper is to report our recent study on the 298–1073 K tensile property. The features and effects of DSA and dynamic precipitation will be emphasized. The term “dynamic precipitation” is used to contrast that in a static process⁽¹⁶⁾.

II. Experimental Procedures

One hot-rolled alloy and two cast alloys, which are designated as Fe-26Mn-9Al-0.9C, Fe-35Mn-9Al-1C and Fe-32Mn-9Al-1C, respectively, were used in this study. Their chemical compositions are listed in Table 1. The two cast alloys, Fe-35Mn-9Al-1C and Fe-32Mn-9Al-1C, were solidified in Y-shaped metal and sand molds, respectively, with dimensions of 30 mm $\times$ 145 mm $\times$ 100 mm in the parallel section. All the three alloys were solution treated at 1323 K for 1 h, then oil quenched. Their optical micrographs after solution treatment are shown in Fig. 1. The two cast alloys are fully austenitic. The hot-rolled Fe-26Mn-9Al-0.9C alloy contains about 4 vol% ferrite, as shown in Fig. 1(a) by the stringers of small grains elongated in the rolling direction. This paper reports the common deformation features of the three alloys.

The solution-treated alloys were machined into tensile specimens 35 mm in gage length and 6 mm in gage di-

[†] Graduate Student, National Cheng-Kung University. Present address: Kirii Machine Manufacturing, Co., Ltd., Tochigi 329-41, Japan.

Table 1 Chemical composition of the alloys used in the study (mass%).

Alloy	Mn	Al	C	Si
Fe-26Mn-9Al-0.9C (hot-rolled alloy)	25.5	8.78	0.86	0.44
Fe-35Mn-9Al-1C (cast in metal mold)	35.2	9.12	1.02	1.2
Fe-32Mn-9Al-1C (cast in sand mold)	32.2	9.43	1.03	1.2

Fig. 1 Optical micrographs of the alloys after solution treatment: (a) Fe-26Mn-9Al-0.9C hot-rolled alloy, (b) Fe-35Mn-9Al-1C cast alloy, (c) Fe-32Mn-9Al-1C cast alloy.

ameter. The tensile tests were performed under constant cross-head speeds from 0.2 to 20 mm/s, namely, under constant initial strain rates from 9.4×10^{-5} to 9.4×10^{-3}

Fig. 2 Flow curves as a function of temperature for the Fe-26Mn-9Al-0.9C hot-rolled alloy at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$.

s^{-1} . The tensile temperatures were varied in the range from 293 to 1073 K. The high temperatures were reached within 20 min by a furnace which was attached to the tensile test machine. The specimens were then held isothermally for 15 min before testing. The temperature fluctuation was within 2 K during the tests.

After tensile testing, the fracture surface of the specimens was examined by SEM. The phases present in the specimens were identified by TEM. The TEM thin foils were taken from the gage length section perpendicular to the tensile direction. In addition, the gage length section and the grip section were examined optically after polishing and metallographic etching with 5% nital. In order to detect static precipitation hardening, Vickers hardness tests were also performed on the grip section where plastic deformation was negligible.

III. Results

Typical flow curves of the test alloys as a function of temperature are similar to those illustrated in Fig. 2. As indicated by the serrated portions of the curves, flow instability occurs in two temperature regimes. The two temperature regimes are more clearly revealed in the Arrhenius plots of $\ln \dot{\epsilon}$ vs. $1/T$ given in Figs. 3 and 4, where $\dot{\epsilon}$ and T are the strain rate and temperature, respectively. According to these Arrhenius plots, the apparent activation energies associated with the onset temperatures of instability in the two temperature regimes are calculated as those listed in Table 2.

Fig. 3 The Arrhenius plots showing the regimes of serrated flow for Fe-26Mn-9Al-0.9C hot rolled alloy. The flow curves with serration are indicated by ■, and the smooth flow curves are indicated by □.

Fig. 4 The Arrhenius plots showing the regimes of serrated flow for the cast alloys. Square symbols □ ■ present the data on the Fe-35Mn-9Al-1C cast alloy, and triangle symbols △ ▲ shows the data on the Fe-32Mn-9Al-1C cast alloy.

Table 2 Activation energies obtained at the onset temperatures of serration.

Alloy	Low temp. regime	High temp. regime
Fe-26Mn-9Al-0.9C	140.4 kJ/mol	182.3 kJ/mol
Fe-35Mn-9Al-1C	138.3 kJ/mol	—
Fe-32Mn-9Al-1C	—	182.9 kJ/mol

Qualitatively, all the three test alloys possess the same temperature dependence of flow stress and elongation. As an illustration, the flow stress and elongation data on the Fe-26Mn-9Al-0.9C hot-rolled alloy at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$ are plotted in Figs. 5 and 6. In order to illustrate the static aging effect, the hardness data obtained from the grip sections of the same alloy after tension are

Fig. 5 0.2% off-set yield stress as a function of temperature for the Fe-26Mn-9Al-0.9C hot-rolled alloy tested at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$. The serrated flow regimes are marked by the lines near abscissa.

Fig. 6 Elongation as a function of temperature for the Fe-26Mn-9Al-0.9C hot-rolled alloy tested at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$. The serrated flow regimes are marked by the lines near abscissa.

shown in Fig. 7. As indicated in Fig. 5, there exists a strengthening effect in the low temperature flow instability regime in which the flow stress is nearly temperature-independent in contrast to the usual monotonic decrease with temperature. As the temperature increases in the high temperature flow instability regime, a more pronounced strengthening effect exists. In parallel to this high temperature strengthening, Fig. 6 depicts a drastic elongation drop, whereas Fig. 7 indicates a static aging effect. When the elongation drops to a minimum, the ten-

Fig. 7 Microhardness of the grip section obtained after high temperature deformation (Fe-26Mn-9Al-0.9C, strain rate: $9.4 \times 10^{-5} \text{ s}^{-1}$).

Fig. 8 Fractographies of the hot rolled Fe-26Mn-9Al-0.9C alloy deformed at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$, showing (a) dimple fracture at 723 K, (b) intergranular fracture at 973 K at which the elongation is the minimum.

specimens undergo intergranular fracture instead of the normally observed dimple fracture. The fractured specimens tested at the two temperatures 723 and 973 K are compared in Fig. 8.

Fig. 9 Optical micrographs showing the precipitation in the deformed gage sections after tested at the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$: (a) Fe-26Mn-9Al-0.9C, 823 K, (b) Fe-26Mn-9Al-0.9C, 873 K, (c) Fe-32Mn-9Al-1C cast alloy, 873 K, where the tensile direction is indicated by the arrows.

In those specimens exhibiting a high temperature strengthening effect and intergranular fracture, Fe_3AlC_x precipitates can be observed in the deformed gage length section. Given in Figs. 9(a), (b) and (c) are the optical micrographs of the gage length sections which were polished and metallographically etched after testing at 823 and 873 K. As illustrated in these micrographs, the precipitates tend to align into sets of parallel lines resembling slip lines on the surface. Such slip-line-like feature is not present in the grip section of negligible strain. It is

also absent in the specimens deformed below the precipitation temperature of Fe_3AlC_x . Figure 9(a) reveals a grain with annealing twins of different $\{111\}$ twin planes. The sets of parallel lines in this grain are parallel to two of the twin planes, indicating that the precipitation takes place preferentially on $\{111\}$ slip planes. The TEM image and diffraction pattern of the precipitates are shown in Fig. 10. The precipitation takes place not only on $\{111\}$ slip planes but also on grain boundaries. A typical example showing grain boundary precipitation is shown in Fig. 11 in which the specimen was tested at 973 K.

Fig. 10 TEM observation on the Fe-35Mn-9Al-1C cast alloy after being strained at 873 K, $9.4 \times 10^{-5} \text{ s}^{-1}$: (a) bright field image, (b) diffraction pattern, (c) dark field image obtained from $[001]$ diffraction of the Fe_3AlC_x .

Fig. 11 Optical micrograph of the gage section after deformation at 973 K, $9.4 \times 10^{-5} \text{ s}^{-1}$, showing grain boundary precipitation.

IV. Discussion

The common deformation behaviors of the hot-rolled Fe-26Mn-9Al-0.9C alloy and the two austenitic cast alloys are reported in the above observations. No remarkable results related to the ferrite phase were detected in the hot-rolled alloy. Therefore, the experimental data can represent the general characteristics of the high temperature tensile properties of austenitic Fe-Mn-Al-C alloys.

1. The DSA of austenitic Fe-Mn-Al-C alloys

All the test alloys exhibit the flow instability which is the general characteristic of dynamic strain aging⁽¹⁷⁾. This instability, indicated by serration in the flow curves (see Fig. 2), occurs in two temperature regimes (see Figs. 2, 3 and 4). At the strain rate of $9.4 \times 10^{-5} \text{ s}^{-1}$, the two temperature regimes are about 430–640 K and 720–870 K for the hot-rolled Fe-26Mn-9Al-0.9C alloy. The flow instability that takes place in two temperature regimes has also been observed in those alloys containing both interstitial and substitutional solute atoms, such as Mn-Cr steels and Ni-Cr stainless steels⁽¹⁸⁾⁽¹⁹⁾.

As listed in Table 2, the activation energy associated with the onset of serration in the low temperature regime is about 140 kJ/mol (33 kcal/mol). This activation energy is close to that for the volume diffusion of interstitial carbon atoms in the austenitic matrix⁽²⁰⁾⁽²¹⁾, implying that the diffusion of carbon should be responsible for the flow instability at low temperatures. The diffusion of carbon has also been suggested to be responsible for the flow instability of many alloys such as Mn-Cr steels⁽¹⁸⁾, Ni-Cr stainless steels⁽¹⁹⁾⁽²²⁾⁽²³⁾ and Hadfield steels⁽²⁴⁾⁽²⁵⁾.

On the other hand, Table 2 shows that the activation energy in the high temperature regime is about 180 kJ/mol (44 kcal/mol). It is about 0.6 to 0.8 the activation energy for the volume diffusion of substitutional alloying elements in an austenitic iron matrix, i.e., 235 kJ/mol (56 kcal/mol) for aluminum and 275 kJ/mol (66 kcal/mol) for manganese⁽²⁰⁾. Since the serration achieved by a short-range diffusion within the dislocation core region

requires the activation energy of about 0.4 to 0.7 that of volume diffusion⁽²⁶⁾⁽²⁷⁾, the above result implies pipe diffusion as a possible mechanism in the high temperature DSA regime. The high temperature DSA regime also falls in the aging temperature regime of Fe_3AlC_x precipitates. Many studies of Ni-Cr stainless steels⁽¹⁸⁾⁽¹⁹⁾⁽²²⁾⁻⁽²⁷⁾ have shown that the DSA at high temperatures is associated with the interaction between chromium and carbon atoms or the precipitation of carbides. Therefore, the differences in the activation energies may also be accounted for by the solute interaction which forms Fe_3AlC_x precipitates.

2. The yield stress

In contrast to the usual experience that yield stress decreases with the rise of temperature, Fig. 5 shows that the 0.2% off-set yield stress is nearly temperature independent in the temperature regime of about 430 to 700 K, which is also the low temperature DSA regime, and then increases to a maximum at the temperatures above 770 K. Also, as shown in Fig. 6, the elongation decreases above about 770 K.

Associated with low temperature DSA, the temperature independence of the yield stress in the temperature range of about 430 to 700 K is accounted for by the strengthening effect due to carbon atmosphere drag. Such behavior is common for the DSA in austenitic alloys⁽¹⁸⁾⁽¹⁹⁾⁽²²⁾⁻⁽²⁴⁾.

At the temperatures of about 770 K and higher, not only the high temperature DSA but also the precipitation of Fe_3AlC_x particles take place. The tensile properties are thus influenced by both the DSA and the carbide precipitation. However, it has been found that DSA has less effect on the strength, work-hardening and ductility of austenitic Mn-Cr steels and Ni-Cr stainless steels⁽¹⁸⁾⁽²²⁾⁽²³⁾. As an implication, precipitation hardening should play the major role in the yield stress and ductility of the Fe-Mn-Al-C alloys in this temperature range. The implication is supported by the microhardness data of the undeformed grip sections in Fig. 7 which reveal that static aging at these temperatures can give rise to pronounced hardening effect.

3. Dynamic precipitation

As pointed out previously, austenitic Fe-Mn-Al alloys can be strengthened by the formation of Fe_3AlC_x after static aging⁽¹⁰⁾⁻⁽¹²⁾. According to the tensile condition at the temperatures above 773 K, the duration of the tensile tests is about 40 min even at the fastest strain rate ($9.4 \times 10^{-3} \text{ s}^{-1}$). In the grip section of negligible plastic deformation, Fig. 7 reveals that this holding period is sufficient to give rise to static aging effect. In contrast to the static aging in the grip section, the gage length section undergoes dynamic precipitation during deformation⁽¹⁶⁾. For the Fe-Mn-Al test alloys, the dynamic precipitation process leads to slip-line-like feature which is formed by aligning the precipitates on {111} slip planes and is not observable in the grip section.

During dynamic precipitation, Fig. 9(a) reveals that

the sets of parallel lines in the grain with annealing twins are parallel to two of the three {111} twin planes which are inclined to the tensile direction. In this grain, no lines are observed on the slip plane which has its plane normal nearly parallel to the tensile direction and hence has the Schmid factor close to zero. As an indication, the precipitation takes place mostly along the active slip planes.

The mechanisms of Fe_3AlC_x precipitation are worth noticing. Many studies have suggested that the precipitation is achieved through spinodal decomposition⁽¹⁰⁾⁻⁽¹²⁾. Heterogeneous nucleation of precipitates on grain boundaries (see Fig. 11) and twin boundaries have also been reported⁽¹¹⁾⁽²⁸⁾. According to Fig. 9, heterogeneous nucleation of precipitates at the dislocations on the active slip planes during tensile straining is proposed. The heterogeneous nucleation has also been revealed by aging the prestrained Fe-26Mn-9Al-0.9C specimens. In the experiment, 5% strain was applied at room temperature, and then the deformed samples were heated at 873 K for 3 h. The results indicate that precipitates with slip-line-like features as those depicted Fig. 9 can be observed in the gage length but not in the grip.

4. Embrittlement

The alloys exhibit minimum ductility at the temperatures between about 773 and 1023 K which are also the aging temperatures leading to Fe_3AlC_x formation. Figures 9(a) and (b) indicate that cracks form along the grain boundaries in the gage section. As shown in Fig. 11, precipitation not only takes place within the grains, but also along the grain boundaries. As the grain boundaries are weak compared with the precipitate-hardened grains, cracks can easily nucleate at those grain boundaries normal to the tensile direction, resulting in the intergranular fracture.

V. Conclusions

The following conclusions are drawn in this investigation:

- (1) All three Fe-Mn-Al-C alloys have similar tensile behavior involving the DSA and dynamic precipitation.
- (2) The DSA exhibits flow instability in the two temperature regimes of about 430 to 640 K and 720 to 870 K.
- (3) The apparent activation energy for the onset of low temperature flow instability is about 140 kJ/mol, suggesting that the low temperature flow instability is attributed to carbon atmosphere drag.
- (4) The apparent activation energy for the onset of high temperature flow instability is about 180 kJ/mol, which is only about 0.6 to 0.8 that associated with the volume diffusion of substitutional manganese and aluminum. The high temperature flow instability may thus be influenced by pipe diffusion and carbide precipitation.
- (5) Associated with carbon atmosphere drag, the 0.2% off-set yield stress is temperature-independent in the low temperature DSA regime.
- (6) The high yield stress at the temperatures above about 773 K is attributed to the strengthening effect by

the precipitation of Fe_3AlC_x .

(7) Grain boundary precipitation plays a major role in the embrittlement and intergranular fracture at the temperatures above about 773 K.

(8) Dynamic precipitation takes place at the dislocations on the active $\{111\}$ slip planes, resulting in the alignment of the precipitates parallel to the annealing twins within the austenitic grains.

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