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The Origin of Serration at High Temperature Deformation in Feeder Pipe Material in CANDU Reactor

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1. Introduction

The yield point is a phenomenon in which the stress decreases at the beginning of deformation near yield stress in mild steel. Cottrell reported that the yield point phenomenon occurs in low carbon steel [1]. He explained by the assumption that the mobile dislocations are pinned by the solute carbon and the stress is decreased when the dislocation is released [2]. Cottrell has investigated in WQ + strain + aging state at 30°C only. It is possible to explain that the aging process has satisfied a condition in which the dissolved carbon moves to the mobile dislocation. This explanation may be possible in a limited condition.

However, these experiments and explanations have long been overlooked as the excluded experiments of various phenomenon like short range ordering that occurs in mild steel.

This study confirms that there is a short range order (SRO) reaction occurring in the water quenched (WQ) mild steel using DSC. The serrations during high temperature deformation is explained by the existence of SRO phase.

2. Experimental

The mild steel rod with 10mm diameter was used. The carbon content is equivalent with SA 106 class B, which is equivalent to feeder pipe material in CANDU reactor. The chemical composition of mild steel is shown in Table 1.

The specific heat (Cp) of mild steel is determined using a water quenched (WQ) from 960°C and a furnace cooled (FC) state.

In order to identify the nature of exothermic reaction during DSC (differential scanning calorimeter), the WQ specimens are scanned in different rate of 10-40k/min. The scan rate variation is a well-known experimental method to determine the activation energy for the exothermic reaction. The activation energy for the ordering reaction is calculated as follows [3];

$$\ln((\alpha_2 T_1^2/\alpha_1 T_2^2) = Q/R(1/T_1 - 1/T_2) \quad (1)$$

where T_1 and T_2 are peak temperatures at scan rate α_1 and α_2 and R is gas constant.

The microstructure is examined by SEM and electron back scattered diffraction (EBSD). The specimens are prepared by grinding and electro-polishing in 10% perchloric acid + 90% methanol solution.

The tensile test was carried out from RT to 500°C under the condition of 6.6×10^{-6}/s.

Table 1. Chemical composition of SS316L (wt. %).

elements	Fe	Mn	Si	N	C	P	S
Composition [%]	Bal.	0.81	0.29	0.004	0.23	0.02	0.006

Table 2. Peak temperature variation with heating rate in water quenched mild steel.

Material condition	Heating Rate (α, ℃/min)	Peak Temperature (Tp, ℃)	1/T [K^{-1}]	α/Tp2
water quenched mild steel	**10**	295.6	0.001758	3.09141E-05
	20	319.2	0.001688	5.69998E-05
	30	333.9	0.001647	8.1409E-05
	40	344.4	0.001619	0.000104886

3. Results and Discussions

The microstructure of FC and WQ mild steel is shown in Fig. 1. The microstructure of FC mild steel is consisted of ferrite and pearlite. The microstructure of WQ mild steel is consisted of ferrite and martensite.

The Cp variations in mild steel are shown in Fig. 2. The exothermic reaction appears at 120-450°C in WQ. This is due to the reduction entropy in WQ mild steel. This means that the ordering reaction occur in mild steel. The nature of the ordering reaction seems to be the short range ordering (SRO) between Fe and C. The amount of released energy (FC-WQ) is calculated as 5 J/g.

The DSC results are shown in table 2. This results are plotted in Fig. 3. The calculated activation energy for the SRO reaction in WQ mild steel, Q ordering is 73kJ/mol. This seems to be a reasonable value since the SRO reaction in mild steel occurs between Fe and C.

The strain-stress curves at 55°C in FC and WQ mild steel are compared in Fig. 4. The FC steel shows a yield point and weak serration at 55°C. The yield point is formed at the beginning of deformation during tensile test in FC state, whereas yield point is not formed in WQ state.

Fig. 4 shows the results of tensile test of WQ and FC immediately at RT and of WQ and FC after holding at 55°C for about 6 days. The FC specimens remained unchanged for 6 days, while the yield strength of WQ specimens became doubled. It can be seen that the WQ specimen changes with time at 55°C. This strengthening effect is due to SRO reaction at 55°C.

In Fig. 2 and 3, the DSC analysis shows that the WQ mild steel exothermic reaction occurs at 150-400°C. This reaction implies SRO reaction in mild steel. On the other hand, the FC mild steel does not generate an exothermic reaction, indicating that the SRO reaction has already been completed. That is, since the FC mild steel has SRO formed basically, the yield point appears by the initial SRO shearing. However, the WQ mild steel does not have an SRO, so yield point does not appear since there is no SRO to be sheared.

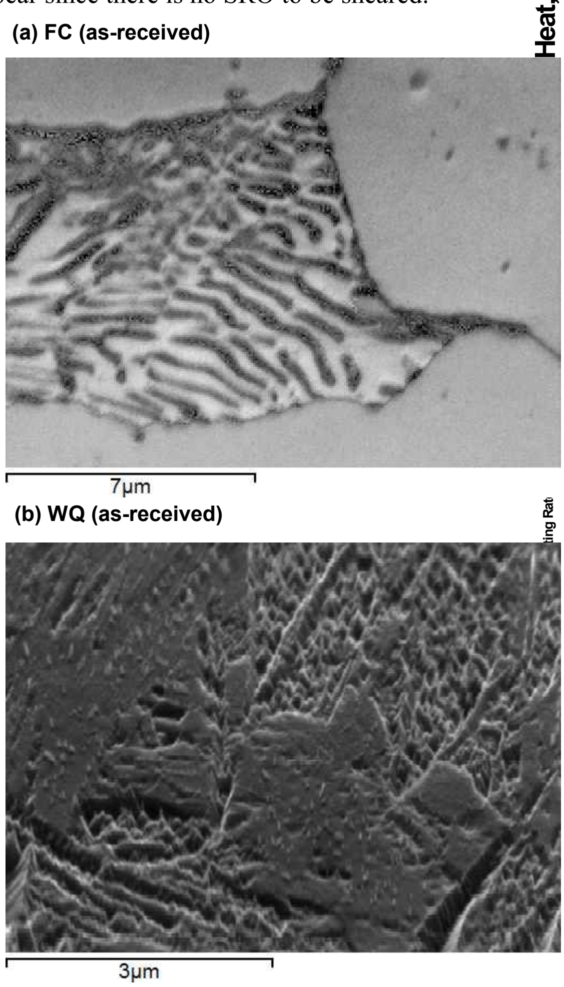

Fig. 1. Microstructure in furnace cooled (FC) and water quenched (WQ) mild steel.

Yield point occurred at the beginning of deformation, whereas the serration is formed during in the middle region of deformation. The reason of yield point and serration seems to be different slightly, since the state of deformation is not same.

The WQ material does not show yield point at all temperature at all. However, the serration occurs 50-160°C. In addition, the yield and tensile strength increases with temperature at below 100°C. It is possible to understand that the deformation behavior at high temperature is influenced by cooling rate significantly in mild steel. It is reasonable to understand that the state of SRO affects the deformation behavior at all temperature in mild steel

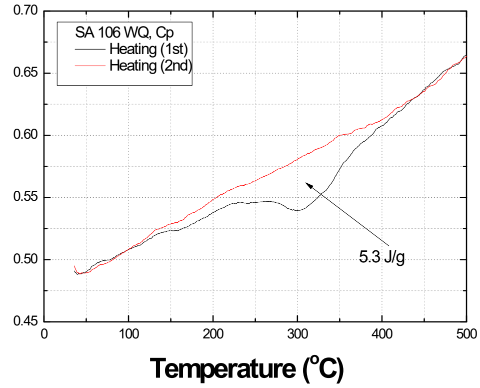

Fig. 2. The specific heat (Cp) variation in WQ and Mild steel.

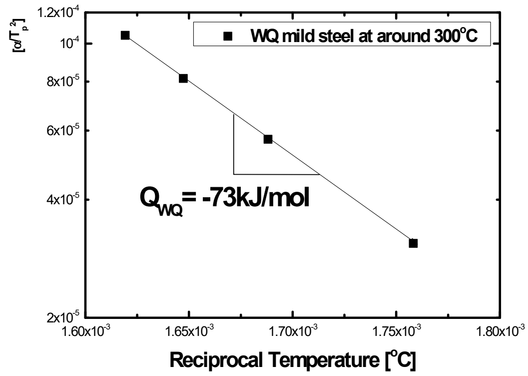

Fig. 3. The activation energy measured by DSC in WQ mils steel.

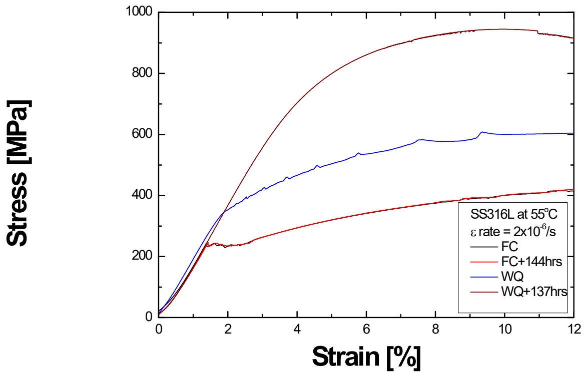

Fig. 4. Strain-stress curves with temperature in FC and WQ mild steel at 55℃.

Although not shown here, the SADP (selected area diffraction pattern) by TEM analysis shows that the FC specimen has an additional faint peak at a position not seen in the BCC structure. This is the conclusive evidence that there is an SRO in the FC-treated specimen that cannot be seen in the BCC crystal structure.

Cottrell seemed unaware of the SRO phenomenon occurring in the Fe-C alloy. In this test, it would have been sufficient to consider the mobile dislocation and solute carbon at the WQ + strain + aging at 30℃ state. Therefore, the specimen with the aging of deformed specimen with mobile dislocation after WQ treatment only showed in yield point. Therefore, it seems reasonable to interpret that the pinning effect of the solute carbon being transferred to the mobile dislocation occurs. However, this is only an interpretation without the fact that the presence of the SRO phenomenon. It is not possible to explain that heat is generated in the process of the pinning of solute carbon to mobile dislocation.

Fig. 4 shows the effect of RT aging on WQ and FC specimens in stress-strain curve. The yield strength of the WQ specimen was approximately doubled by RT aging. On the other hand, FC specimens hardly change. That is, while the WQ specimen is maintained at 55°C for 6 days, it seems that a very fine SRO is formed. This is the reason why the yield strength in WQ aged at 55°C is doubled. On the other hand, since the SRO is already formed in the FC specimen, the aging for 6 days of 55°C has no effect. Fig. 2 shows that the exothermic reaction does not appear in the FC specimen. The WQ specimen is disordered and no SRO image is formed in SADP results, but the SRO is formed during DSC analysis and the heat is generated. On the other hand, FC specimens do not undergo exothermic reaction in DSC analysis, thus aging at room temperature causes little change in strain behavior.

4. Conclusions

1. The occurrence of SRO reaction in mild steel was confirmed by DSC and TEM analysis.
2. The exothermic (SRO) reaction cannot be explained by the fact that the solute carbon pins mobile dislocations in mild steel.
3. The formation of serration during tensile test can be explained by the load drop due to the shearing of SRO and the load increase due to formation of SRO by strain induced ordering (SIO) between Fe and C in mild steel.

Acknowledgements

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