Egypt. J. Sol., Vol. (16), No. (1), (1993)

NATURE OF INTERACTION BETWEEN LATTICE DISLOCATIONS AND GRAIN BOUNDARIES DURING HIGH-TEMPERATURE CREEP OF IRON

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Abstract

Specimens of Iron of commercial purity in the alpha-phase, were tested for creep at 773 K under applied stresses that ranged from 100 to 220 MPa till fracture. Two of these crept specimens were then selected for the electron microscope examination of structure. The grain boundary movements, misorientations, absorption of lattice dislocations and their relative sliding against each other were observed. The contribution of the grain boundary movements and their enhancement of the process of creep and the creep rate sensitivity parameters were considered.

Introduction

Extensive studies have been made previously on the effect of changes of structure of grain boundaries on their dynamic properties [1-4]. It is accepted that grain boundaries change their structure during important metallurgical phenomena, especially during creep deformation [1]. Unfortunately, little knowledge is know about such grain boundary changes of structure. It seems of importance to study these structural changes in order to understand clearly such related metallurgical processes. It was observed [3,4] that random boundaries change into an almost oriented boundaries by absorbing a number of lattice dislocations during sliding in high temperature creep of aluminium. The results [3,4] were discussed on the basis of the moving lattice dislocations and their absorption at grain boundaries. Other workers [5,6] estimated a relation between foreign atoms segregation and grain boundary precipitations and their combined effect on creep properties. Also, some evidence were given on the interaction of the moving boundaries with impurity contents or solute atoms [7,8].

In this work, a trail to find an evidence, using the electron microscope, relating the structure of grain boundaries and the lattice dislocations is carried out on specimens of technically pure Iron (Armco of purity 99.5%) after being subjected to creep tests at 773 K under applied stresses of 122 and 220 MPa.

Experimental

The material used in this work is Iron of purity 99.5%. The specimens were first subjected to creep tests at 773 K till fracture. The applied stress ranged from 98 to 220 MPa. A creep machine operated at wide range of applied stresses was carefully adopted for this purpose. The temperature was maintained constant to within ±1K.

Foils for transmission electron microscopy were prepared from two of these specimens that were crept at 122 and 220 MPa. They were first spark cutted to small plates of about 0.5 to 0.8 mm thick. The cutting machine uses a fine wire of copper and a voltage difference of about 300 V was applied between the specimen and the moving wire. The plates were then attached to a flat surface holder of steel by means of Kanagom, to be polished and thinned to about 0.1 mm. A combined chemical and electrolytically polishing was used to prepare the platelets to the electron transmission examination. For chemical etching a solution of 28 cm^3 H_2O_2 (30%) with 2 cm^3 of conc. Hydrofloric acid was used. For electrolytic polishing the platelets were fixed to a holder connected to a D.C. circuit (with 20 V as an anode). The electrolyte was 200 gm orthophosphoric acid mixed with 100 gm chromic oxide at a temperature of at least 333 K.

The thin foils after such fine polishing and etching were then investigated by the Hitatchi HU 11 A transmission electron microscope at the magnification of 5000 to 15000.

Results :

Fig. (1) represents a set of creep curves of Iron taken at 773 K, under applied stresses changed regularly from 98 MPa to 220 MPa.

The logarithmic relation between the steady state creep rate ε_s and the applied stress σ, at the temperature of 773 shown in Fig. 2, yield two parts of straight lines, corresponding to the range of the applied stress. The slopes of these lines represent the applied stress sensitivity parameter of the steady state creep rate.

$$m' = \left(\frac{\partial \ln \varepsilon_s}{\partial \ln \sigma}\right)_T$$

It takes the value of about 6 Si% at the stress range around 120 MPa, and reaches to 20 at the higher stress range around 220 MPa.

The electron microscope photograph in the section in Fig. 3 illustrates a randomly oriented network of dislocations. At this part of specimen, the increased density of these partially mobile dislocations are mainly due to the higher level of applied stress (220 MPa). The effect of further duration of stress will lead these dislocations to move inside the lattice and orient themselves in the direction of stress, and to take part in creep strain till reaching the neighbouring boundaries of grains where they were absorbed.

Fig. (4) illustrates one of the configurations between lattice dislocations and grain boundaries. It shows clearly a fine interaction between dislocation lines and their absence or at least their disappearance at the boundaries. The beginning of formation of new subboundaries were also observed. The section at this part of structure is for the low applied stress level (122 MPa).

At the stress 220 MPa, the structure of grains at the boundaries is strongly affected. This configuration is clearly observed in the micrograph section in fig. 5. At the boudnaries, some new

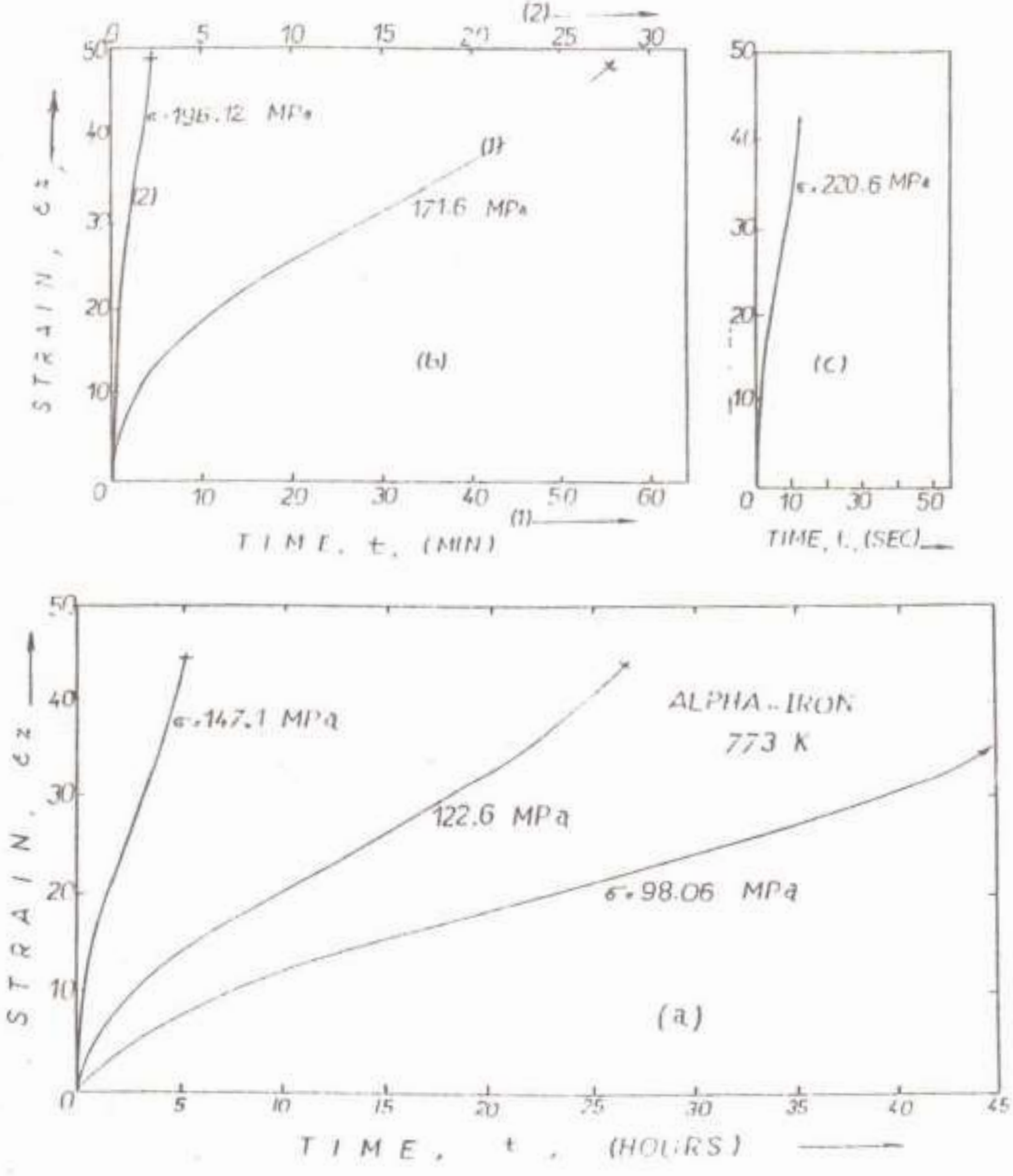

Fig. (1) Typical creep curves for specimens of Iron tested at 773 K under different applied stresses.

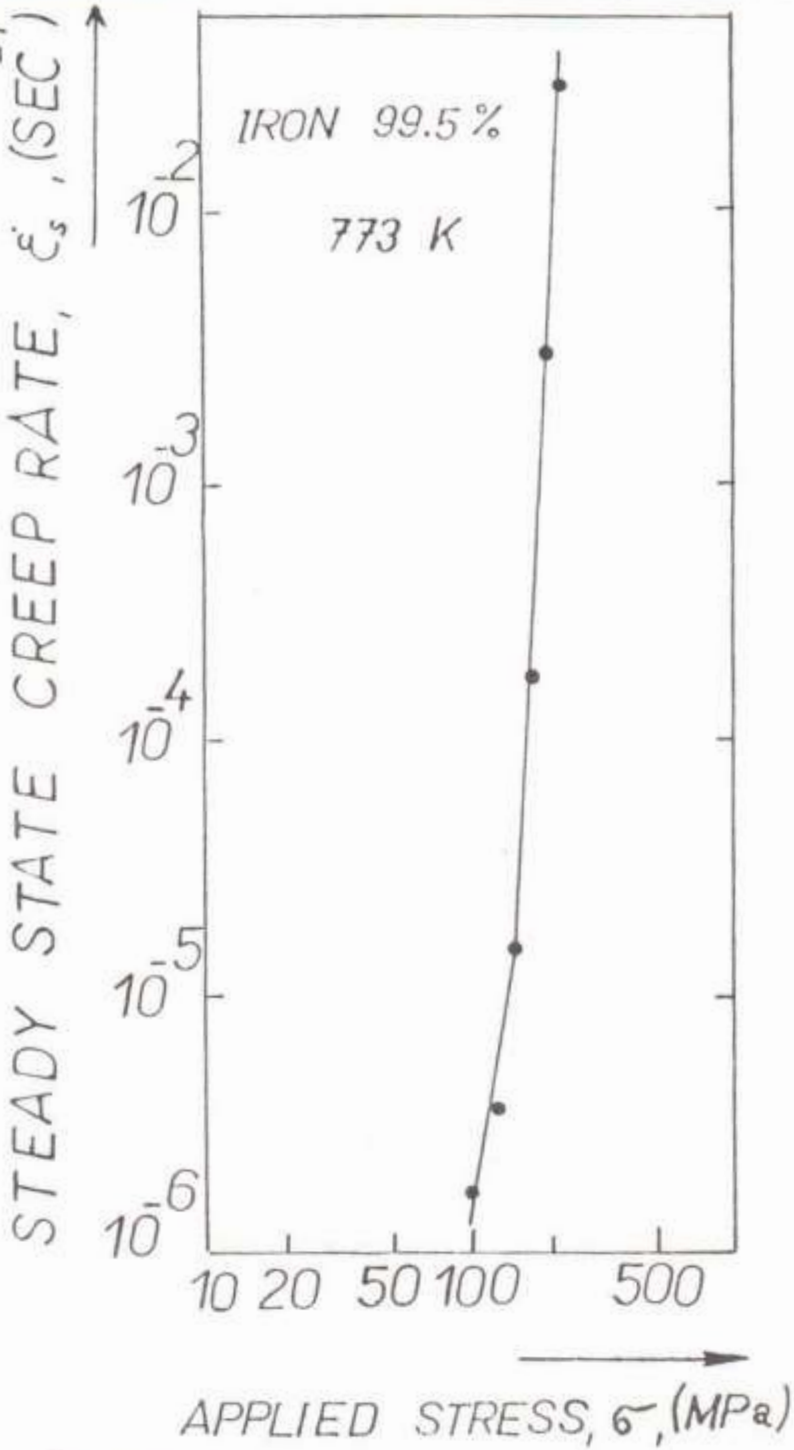

Fig. (2) Logarithmic relation between the steady state creep rate ε_s and the applied stress σ of the Iron samples tested at the temeprature 773 K under diffrent applied stresses.

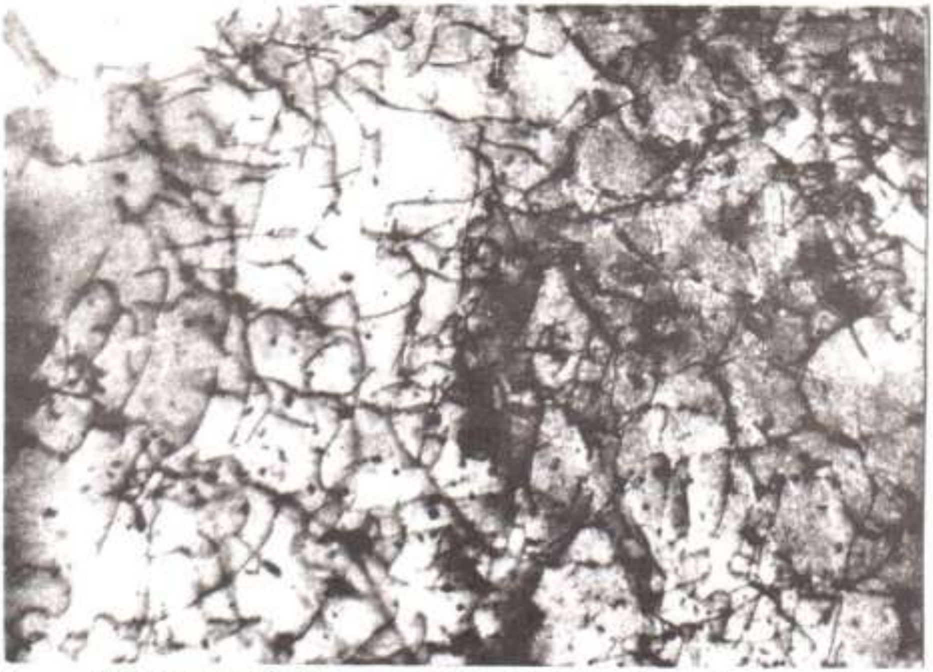

Fig. (3) **Electron micrograph of a section in the specimen that was affected by the higher stress** showing the increased dislocatioin density. **The creep test was carried** under the applied stress 220 MPa at 773 **K. The mangificantion is** 12000 x.

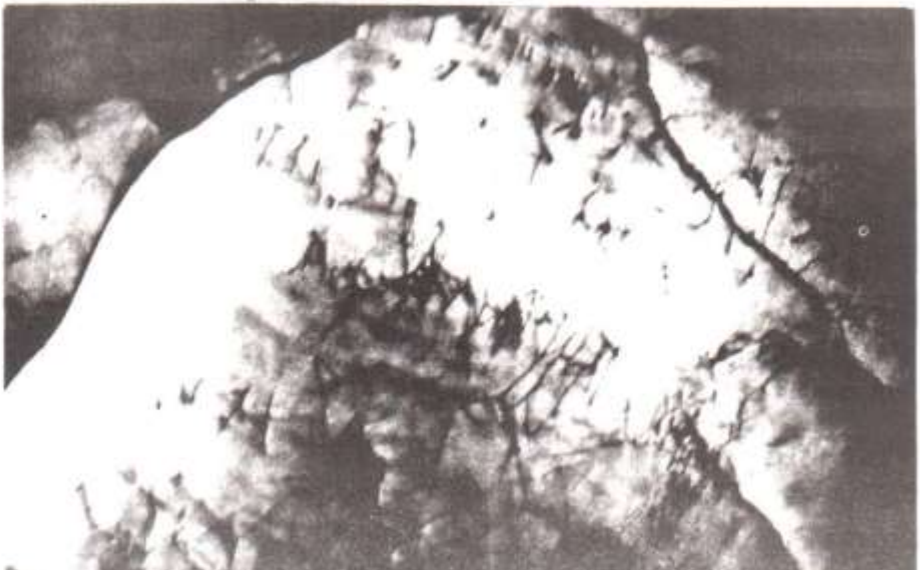

Fig. (4) **Micrograph** showing the interaction between the lattice dislocatioins and **grain** boundaries at a section of the specimen that was tested **under 122 MPa at** 773 K. The magnificatioin is 9000 x orientations, separation and void formation are quite clear, which let us suppose that they most probably add something new to the dynamics of creep strain besides the role of the moving dislocations.

The most affected configurations of structure, are those taken from parts of specimen near the surfaces of fracture as shown in Fig. (6) (a & b). The changes in shape and structure at the boundaries are clearly identified. Such severe changes confirm the role of grain boundary movements in the plastic deformation phenomenon. These two sections are taken from the specimen that was creep tested under 220 MPa at 773 K.

Discussion

The changes in grain boundary shape and structure during creep in different metals and alloys had been widely investigated before [9-12]. It is generally acceptable that grain boundary movements play an important role in creep and in other related metallurgical processes. The present work is a trail to study this phenomenon using the electron microscope. Observations on different sections taken from parts of specimens which were firstly creep examined till fracture under applied stresses of 122 MPa and 220 MPa at 773 K were carried out on pure Iron.

The steady state creep rate results suggest that increasing the applied stress from about 100 to 220 MPa at the temperature or 773 K, change the orientation and generally the shape and structure of grain boundaries to move and act together with the changes in the direction of the moving free dislocations during the process of creep. The absorption of the moving lattice dislocations at the boundaries allow the grains to orient themselves and slide against each other, and accordingly cooperate to accelerate the process of creep. That is why the increase in the values of the applied stress sensitivity parameter m' of the steady state creep rate especially at the higher levels of the applied stress where the density of the free dislocations increases depending on the relative increase of the applied stress. It is most

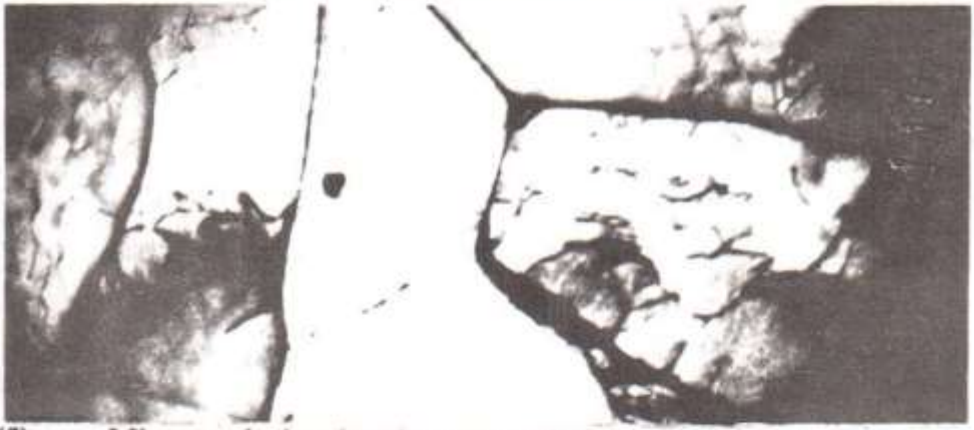

Fig. (5) Micrograph showing the structural changes at the grain boundaries for the specimen that was affected by the higher applied stress. The magnification is 9000 x.

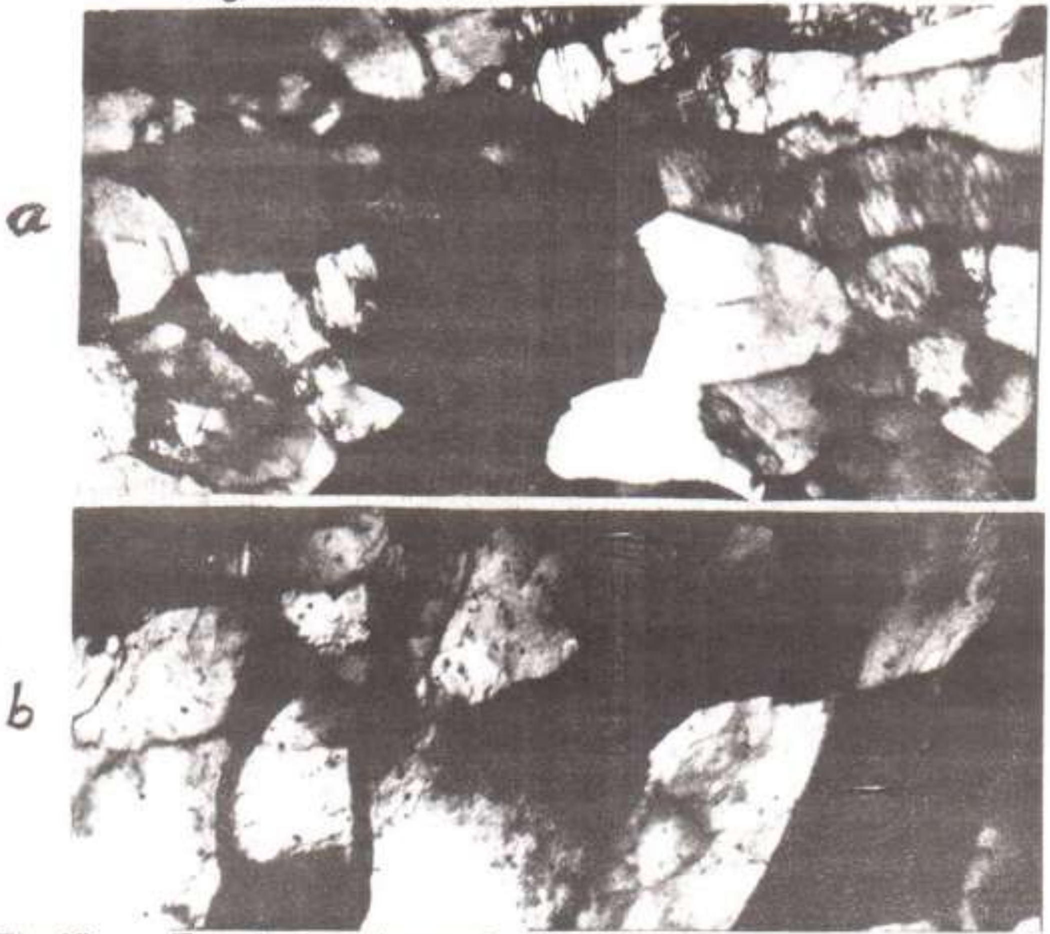

Fig. (6) Two electron micrographs (a) and (b) for two sections at diffeerent parts in the specimen (220 MPa at 773 K), showing the grain boudnaries slidings and change of shape. The magnification are (a) 9000 x, (b) 12000 x.

probable that grain boundary movements is the main factor that arise especially at higher stress levels and contribute to creep strain. The electron micrographs that were obtained in the figures from 3 to 6 show clearly and confirm this supposition as follows:

a) The increased density of dislocations in the section in Fig. 3 characterizes the relatively higher level of applied stress (220 MPa) which when still applied for long duration, these dislocations will depart and move till they reach the boundaries of the surrounding grains where they are absorbed or redistributed.

b) At relatively lower levels of applied stress (122 MPa) the structure changes due to the absorption of lattice dislocations at the boundaries (section in Fig. 4) can be detected from the depth or the darker margins of the boundary edges. The beginning of formation of new subboundaries can also be observed.

c) At the higher level of applied stress (220 MPa) the random boundary edges which changed into an almost exact oriented edges at parts of the specimen shown in fig. 5 are therefore considered to be the cause of the increasing creep strain and the amount of slidings.

d) The severe interaction and the combined role between dislocations and grain boundaries to reorient the whole structure in the direction of stress, and accordingly its role in creep strain are quite clear in the two sections of Fig. 6.

Conclusion

The sensitivity parameter of the steady state creep rate is considered to assist the state of dislocations dynamics as responsible for the creep strain. The increase in the moving dislocation densities on the lattice slip planes as a direct result of increase in the external stress is responsible for the value of the sensitivity parameter m'.At certain increasing stress, the increase of m' is supposed most probably to originate from the grain boundary movements, the phenomenon which is confirmed by the photographs taken from the electron microscope examination.

Acknowledgment

The author wishes to express his sincere thanks to Prof. Dr. J. Cadek fromt he Institute of Physcial Metallurgy of Czechia Academy of Science of Brno of the Republic of Czechia, for his valuable guidance in the electron microsocpe work.

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