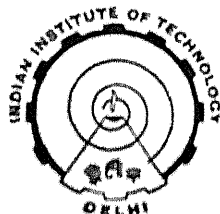


KINETICS AND PLATE DIMENSIONS OF ISOTHERMALLY FORMED MARTENSITE IN AN Fe-Ni-Mn ALLOY

by
GAUTAM GHOSH

Submitted
in fulfilment of the requirements
for the degree of
DOCTOR OF PHILOSOPHY



Department of Applied Mechanics
INDIAN INSTITUTE OF TECHNOLOGY, DELHI
1985

CERTIFICATE

This is to certify that the thesis entitled "Kinetics and Plate Dimensions of Isothermally Formed Martensite in an Fe-Ni-Mn Alloy" by G. Ghosh submitted to the Indian Institute of Technology, New Delhi (India) for the award of the degree of Doctor of Philosophy in Applied Mechanics Department is a record of bonafide research work carried out by him under my supervision and guidance. The thesis work, in my opinion, has reached the standard fulfilling the requirements for the Doctor of Philosophy Degree. The research report and results presented in this thesis have not been submitted in part or in full to any other University or Institute for the award of any degree or diploma.

V. Raghavan
23/9/85
(V. Raghavan)
Professor

Department of Applied Mechanics
Indian Institute of Technology
New Delhi-110016 (India)

ACKNOWLEDGEMENTS

It is a great pleasure to express my deepest sense of gratitude to my supervisor and teacher, Prof. V. Raghavan, for his invaluable guidance, help, suggestions and criticism throughout this work.

Sincere thanks are due to Dr. R.K. Pandey for getting my sample analyzed from International Combustion Ltd., while he was in U.K. Special thanks are due to Dr. A.N. Kumar for his help and cooperation.

I like to extend my gratitude to Dr. S. Ray, Asst. Prof., Dept. of Metallurgical Engineering, Dr. K.C. Mittal, Director, Mr. N.K. Saini, Engineer-in-charge, University Services and Instrumentation Centre, University of Roorkee, for their kind help and cooperation in TEM work.

I am grateful to Mr. Prem Singh, Mr. Onkar Chand, Mr. Bharat Singh and Mr. A.K. Goyal for their assistance and cooperation during various phases of experimental work.

Finally, thanks are also due to Mrs. Madan who painstakingly typed the manuscript.

Date: 23/7/85


(G. Ghosh)

ABSTRACT

The kinetics of isothermal martensitic transformation is influenced by the test temperature, a superimposed elastic stress, prior plastic strain of the austenite and the grain size of austenite. The isothermal mode of transformation provides an ideal opportunity to measure the plate dimensions on a firm basis, as the reaction temperature can be controlled accurately and also the transformation rate is measurable as a function of time. In the present work, the isothermal kinetics as well as the plate dimensions have been studied as a function of reaction temperature, a superimposed elastic stress, grain size and prior plastic strain of the austenite.

An Fe-23.2Ni-2.3Mn alloy is used here for all experiments. The experimental procedures include homogenization, rolling and wire drawing, austenitization, doping, plastic deformation of austenite at room temperature, optical and electron metallography. The virgin austenite is transformed to martensite at nine isothermal test temperatures from -70°C (203K) to -196°C (77K). The effect of a superimposed elastic stress field has been studied at three test temperatures: -85°C (188K), -143°C (130K) and -196°C (77K). The plastically deformed specimens are transformed at -85°C (188K), -100°C (173K), -140°C (133K) and -196°C (77K). The kinetics of the transformation is followed by measuring the electrical resistance of the samples by means of an Autobalance Universal Bridge. The change in resistance is calibrated in terms of the

volume fraction of martensite determined by point counting. The microstructures have been characterized using the optical microscope as well as the TEM. The Fullman method has been used extensively to evaluate the mean dimensions of the martensitic plates. The fraction of the partially transformed grains (G_g) and the extent of plate association (EPA) are obtained by areal and lineal analysis respectively.

The alloy exhibits a typical C-curve behaviour with the nose of the C-curve around -110°C (163K). The kinetic parameters such as the autocatalytic parameter and the activation energy for nucleation are obtained by using the Raghavan-Pati-Cohen approach. The autocatalytic parameter increases slowly but continuously with decreasing reaction temperature. The activation energy for nucleation decreases with decreasing temperature. The computed transformation curves obtained by the above approach agree well with the experimentally observed transformation curves upto a significant extent of transformation. For a particular test temperature, the reaction progresses by a combination of the spreading out of clusters of plates and filling-in of the pockets, both occurring simultaneously. The mean semithickness-to-radius ratio ($\overline{c/r}$) is found to increase with increasing martensitic fraction at any test temperature, but it decreases with decreasing temperature for a given martensitic fraction. The initial $(\overline{c/r})_i$ ratio i.e. $(\overline{c/r})$

at $\sim 0\%$ martensite decreases by a factor of 2, as the test temperature decreases from -70°C (203K) to -196°C (77K). This indicates that a fraction of the transformational strain energy is to be released which decreases with decreasing reaction temperature. This fraction estimated to be about 0.50 at -70°C (203K) and 0.04 at -196°C (77K).

The effect of a superimposed elastic stress on the transformation kinetics and the plate dimensions have been studied by applying various levels of uniaxial tensile stress at three test temperatures. At a given test temperature, with the increasing level of applied stress, the initial nucleation rate and the autocatalytic parameter increase, whereas the activation energy for nucleation decreases. However, the increase in nucleation rate or the decrease in activation energy is more at higher temperatures than at lower temperatures. Three linear equations with different slopes are obtained between the activation energy for nucleation and the total driving force at the three test temperatures, where the elastic stress experiments are carried out. From these slopes, the activation volumes for the dislocation motion associated with the nucleation process are obtained. The activation volume for nucleation is found to decrease by a factor of 15 as the test temperature decreases from -70°C (203K) to -196°C (77K). The activation energies for nucleation at

different stress levels have been calculated by applying the concept of thermally activated motion of dislocations. Also, the activation energies for nucleation at different test temperatures (where no stress was applied) are calculated from the Olson-Cohen model. In most of the cases the calculated and the observed activation energies are in good agreement. The $(\bar{c}/\bar{r})$ ratio is found to increase with the increasing level of applied stress at any test temperature. The calculated (c/r) agrees reasonably well with the experimental $(\bar{c}/\bar{r})_1$.

The effect of prior plastic strain of austenite on the transformation kinetics and plate dimensions have been studied at various plastic strains from 0.005 to 0.05. For any test temperature, the initial nucleation rate and the autocatalytic parameter decrease, whereas the activation energy for nucleation increases. At higher temperatures, the decrease in the initial nucleation rate or increase in the activation energy is more than at lower test temperatures. By taking into account the increase in flow stress due to work hardening at the corresponding test temperatures and plastic strains, the activation energies for nucleation are calculated with the help of the temperature dependence of the activation volume. As before, the assumption is that the nucleation of martensite involves a process of dislocation motion. The calculated and the experimental activation energies are in satisfactory agreement. The work hardened

matrix is found to restrict the growth of the martensite plates. The $(\bar{c}/r)$ ratio decreases with the increasing degree of prior plastic strain at any test temperature and martensitic fraction. The (c/r) ratio is calculated as a function of prior plastic strain and test temperature by taking into account the increase in the flow stress due to work hardening. The calculated (c/r) and the experimentally observed $(\bar{c}/r)_1$ values are found to be in good agreement.

Using three grain sizes 0.019 mm, 0.048 mm and 0.09 mm (mean linear intercept), the effects of grain size on the transformation kinetics and plate dimensions have been studied. Both the activation energy for nucleation and the autocatalytic parameter are found to be independent of grain size at any test temperature. This confirms that the autocatalytic parameter is uniquely related to the driving force. The observed nucleation rates are found to increase by a factor of 2-3 with increasing grain size from 0.019 mm to 0.09 mm. At any reaction temperature and martensitic fraction f , the fraction of the austenitic grains G_g partially transformed to martensite increases with increasing grain size. Also, for any given grain size and f , G_g increases with decreasing reaction temperature. At any test temperature, the extent of plate association (EPA) is independent of f and grain size. However, EPA is found to increase with decreasing test temperature and in the presence of a superimposed elastic stress but decreases in the prestrained austenite.

CONTENTS

	Page No.
Acknowledgements	
Abstract	
List of Tables	1
List of Figures	iv
List of Symbols	xv
CHAPTER 1 INTRODUCTION	1
1.1 General Features of Martensitic Transformations	1
1.1.1 Different Kinetic Modes	1
1.1.2 Morphology and Substructure of Martensite	3
1.1.3 The Crystallography of Martensite	5
1.1.4 The Thermoelastic Behaviour	6
1.2 The Isothermal Kinetics	8
1.2.1 Quantitative Models for the Isothermal Kinetics	8
1.2.2 Factors Influencing the Isothermal Kinetics	11
1.2.3 Correlations between the Kinetic Modes	13
1.2.4 Models for Nucleation	13
1.2.5 Activation Energy Dependence on Driving Force	15
1.3 Measurements of the Dimensions of Martensitic plates	16
1.3.1 The Fullman Method	16
1.3.2 The Smith-Guttman Method	17
1.3.3 Previous Results on Plate Dimensions	18
1.4 Aim and Scope of the Present Investigation	19

		Page No.
CHAPTER 2	EXPERIMENTAL PROCEDURES	20
2.1	Thermomechanical Treatment, Material Characteri- zation and Sample Preparation	20
2.1.1	Homogenization	20
2.1.2	Chemical Analysis	20
2.1.3	Rolling and Wire-Drawing	21
2.1.4	Austenization and Doping	22
2.1.5	Samples for Various Experiments	23
2.2	The Isothermal Runs	24
2.3	Metallographic Techniques	25
2.3.1	Polishing and Etching	25
2.3.2	Optical Microscopy	26
2.3.3	Transmission Electron Microscopy	26
2.4	Quantitative Metallography	27
2.4.1	Determination of Austenitic Grain Size	27
2.4.2	Determination of Volume Fraction of Martensite	29
2.4.3	Measurement of Plate Dimensions and Average Volume of Martensitic Plates	30
2.4.4	Determination of the Fraction of Partially- Transformed Grains	32
2.4.5	Determination of Austenite-Martensite and Martensite- Martensite Interfacial Area per Unit Volume	32
2.5	Plastic Straining of Austenite	33
CHAPTER 3	EFFECT OF THE ISOTHERMAL REACTION TEMPERATURE	35
3.1	Introduction	35
3.2	The Isothermal Transformation Kinetics	36
3.2.1	The Experimental Transformation Curves	36

3.2.2	Variation of the Mean Volume of the Plates with Martensitic Fraction	40
3.2.3	The Computed Transformation Curves	44
3.2.4	The Measured and the Computed Nucleation Rates	49
3.2.5	The Autocatalytic Parameter and the Activation Energy	52
3.2.6	Discussion	56
3.3	Dimensions of the Martensitic Plates	57
3.3.1	Measurements and Computation of the Plate Dimensions	57
3.3.2	Variation of $\bar{c}$ with Martensitic Fraction and Reaction Temperature	63
3.3.3	Variation of $\bar{r}$ with Martensitic Fraction and Reaction Temperature	66
3.3.4	Variation of $(\bar{c}/\bar{r})$ Ratio	66
3.3.5	Factors Influencing the $(\bar{c}/\bar{r})$ Ratio	70
3.3.6	Estimation of the Plastic Accommodation ^m from the Measured $(\bar{c}/\bar{r})$ Ratios	73
CHAPTER 4	ISOTHERMAL TRANSFORMATION UNDER A SUPERIMPOSED ELASTIC STRESS	84
4.1	Introduction	84
4.2	The Transformation Kinetics	86
4.2.1	The Experimental Transformation Curves	86
4.2.2	The Computed Transformation Curves	90
4.2.3	The Initial Nucleation Rates	91

4.2.4	The Autocatalytic Parameter	96
4.2.5	Comparison of Some of the Present Observations With Earlier Results	99
4.3	The Temperature Dependence of $\Delta g - \Delta W_a$ Relationship	103
4.3.1	The $\Delta g - \Delta W_a$ Relationship	103
4.3.2	The Temperature Dependence of the Activation Volume	105
4.3.3	Calculation of the Activation Energy Under a Superimposed Elastic Stress	107
4.3.4	The Critical Step in Nucleation	109
4.4	Measurements of the Plate Dimensions	119
4.4.1	Variation of Plate Dimensions with Martensitic Fraction and Applied Stress	119
4.4.2	Calculated (c/r) Ratio under an Applied Stress	126
CHAPTER 5	ISOTHERMAL TRANSFORMATION WITH A PRIOR PLASTIC STRAIN OF AUSTENITE	134
5.1	Introduction	134
5.2	The Isothermal Transformation Kinetics	136
5.2.1	The Experimental Transformation Curves	136
5.2.2	The Computed Transformation Curves	141
5.2.3	The Initial Nucleation Rates	147
5.2.4	The Autocatalytic Parameter	148
5.2.5	The Observed and the Calculated Activation Energies	150