AUTOMATIC AJUSTMENT METHOD TO FIT $T \cdot \log(T/\tau_0)$
SCALING FOR TIME EVOLUTION THERMAL DEPENDING
PROPERTIES OF NANOCRISTALLINE IRON

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Summary. In many forming processes the evolution of the material properties evolves with
time and changes as temperature varies, forcing the use of a time-dependent
thermomechanical computational model to simulate those processes. If the property under
study could be described as a collectivity of microentities jumping over a certain distribution
of energy barriers from one metastable state to another, then the whole behaviour of the
material property follows a $T \cdot \ln(T/\tau_0)$ scaling. The use of $T \cdot \ln(T/\tau_0)$ as an integration variable
could be employed to simplify the computational procedure. Also this scaling description
allows to extrapolate the time evolution behaviour at times (or speeds) that are experimentally
inaccessible. From this scaling it is also possible to determine the attempt period $\tau_0$, needed by
the computational model. All the properties described by the Arrhenius Law could be scaled
by this method.

In the present work the results of applying this scaling method to the hardness of a
nanocrystalline iron powder with several annealing treatments are presented, and an algorithm
for automatic adjustment of a $T \cdot \ln(T/\tau_0)$ scaling is tested.

INTRODUCTION

There is a broad variety of physical systems that show a time-dependent behaviour of some
of their physical properties because of the existence of energy barriers, which separate local
minima corresponding to different equilibrium states of the system. In recent decades,
considerable effort has been devoted to understanding the relaxation phenomena in systems
with different degrees of disorder, such as: high temperature superconductors[1] in which
vortices are submitted to pinning by defects and dislocations, and where the application of a
magnetic field creates metastable states in the vortex lattice; small magnetic particle
systems[2] with a volume distribution and randomly orientated easy axes, which show
blocking phenomena depending on the experimental time windows, and hardness and grain
size time evolution of heat treated single phase materials[3] were atoms and dislocations are
submitted to pinning. The existence of a scaling that allows to plot all the relaxation data
obtained at different temperatures in a unique master curve would make possible to cover a large number of time decades, due to any change in the temperature of the system corresponds to a change in the time scale of the relaxation.

The simplest case for the scaling law of a relaxing system governed by thermally activated processes is the so called $T\cdot \ln(t/\tau_0)$ master curve\(^\text{[4]}\). Due to there is not a known function for fitting, all approximation to the master curve must be subjective and hence the optimal value changes when the operator is changed. In the present work, a not subjective scaling method is presented and its validity is shown scaling the hardness evolution of nanocrystalline iron powder with several annealing treatments.

**RELAXATION CURVES**

The variation of a property for the assembly of weak (or non) interacting entities with a distribution of energy barriers $f(E)$, which are overcome by thermal fluctuations, according to the Arrhenius Law, is given by\(^\text{[2]}\):

$$\Delta P(t) = \Delta P_0^\infty \cdot \int_0^\infty dE \cdot p(t,E) \cdot f(E)$$

(1)

where $P$ is the property under study, $\Delta P_0^\infty$ is the total variation of the property between the initial and the equilibrium state, $E$ is the energy barrier height and the function $p(t,E)$ is given, according to Neel’s theory\(^\text{[5]}\), by:

$$p(t,E) = e^{-\frac{E}{k_B T} - \frac{E}{k_B T_0} \exp\left(-\frac{E}{k_B T_0}\right)}$$

(2)

where $T_0$ is the attempting characteristic time and $k_B$ the Boltzmann constant.

Taking into account that $p(t,E)$ varies abruptly from 0 to 1 as the energy barrier $E$ increases for a given time $t$, it is usual to simplify it by a step function with the discontinuity in $E_c(t)$, the inflexion point of $p(t,E)$, i.e.:

$$E_c(t)=k_B T \cdot \ln(t/\tau_0)$$

(3)

Then, equation (1) could be rewritten as follows:

$$\Delta P(t) = \Delta P_0^\infty \cdot \int_{E_c(t)}^\infty dE \cdot f(E)$$

(4)

where the only parameter of the evolution is the scaling variable $T\cdot \ln(t/\tau_0)$.

The validity of the scaling is determined by the validity of the step approximation, which is acceptable as long as the width of $p(t,E)$ was small compared to the width of the energy barrier distribution, $f(E)$\(^\text{[4]}\). This assumption is usually certain when, under experimental conditions, the time evolution of the system is clear enough.

**FITTING METHOD**

Nevertheless $f(E)$ and its integral are unknown, both must be quite soft functions far from...
first order transitions. In this case, the integral can be locally approximated by its Taylor series, and this series estimated by a local spline. Assuming those premises, an error function \( F(\tau_0) \) could be calculated by:

\[
F(\tau_0) = \sum_i \left[ H_i - S(\frac{T_i \cdot \text{Log}\left(\frac{t_i}{\tau_0}\right)}{2})\right]^2
\]

where \( H_i, T_i \) and \( t_i \) are the experimental hardness, temperature and time (respectively) of the sample \( i \), and \( S(x_i) \) the local spline at the \( i \) point. As the order of \( T\cdot\text{Log}(t/\tau_0) \) points depends on the \( \tau_0 \) value, these points must be sorted each time that \( F(\tau_0) \) is computed.

Using a standard numerical minimization algorithm, a local minima of \( F(\tau_0) \) could be found. Running the procedure from several random distributed starting points, the global minimum can be reached, and deuce the \( \tau_0 \) optimal value.

**EXPERIMENTAL RESULTS**

The powder was hardened in a planetary ball mill using the technique known as Ball Milling (BM). Within each grinding bowl were introduced 6.00 g of iron powder plus 1.2 wt% of ethylene-bis-Stearamide (EBS), 40 chrome steel balls of 10 mm of diameter. The weight relationship between balls and iron powder was 27:1. An argon overpressure applied to the recipients was used to prevent the powder oxidation during the milling.

The mill rotational speed was set at 160 rpm, with a 30 minutes running cycle followed by 30 minutes of stop period to prevent excessive heating of the powder. After 52 hours of effective working time, a 5 \( \mu \)m particle size and 8 nm crystal size powder was obtained[6]. Five series of 10 samples were prepared with the iron powder. Each sample was prepared with \( \sim 0.1 \) g of iron powder sealed in a glass tube under He atmosphere to prevent oxidation. Those samples were thermal treated at different temperatures (between 450 °C and 700°C) during different time (ranging from 1h to 2 days). Eleven Vickers indentations HV0.01 (ISO 6507-1) were carried out in each sample and the average value was taken as hardness value.

After that, the fitting procedure were applied with a cubic spline with 13 points (from i-6 to i+6 point) and reporting an attempting period \( \tau_0 = 1.76 \cdot 10^{-5} \) s. In figure 1 the hardness of samples are plotted vs \( T\cdot\text{Log}(t/\tau_0) \) for this value, showing a good arrangement of the points.

![Figure 1: Scaling plots and fitting curves of nanocrystalline iron powder for \( \tau_0 = 1.76 \cdot 10^{-5} \) s.](image)
CONCLUSIONS

The $T\cdot \ln(t/\tau_0)$ scaling can reduce the cost of mechanical characterization of materials by the reduction of experimental points required to be measured.

The $T\cdot \ln(t/\tau_0)$ scaling could reduce the computational effort of simulations by the reduction of variables involved in calculus of mechanical properties.

The $T\cdot \ln(t/\tau_0)$ scaling can extrapolate results to time (or speed) experimentally unreachable, by a single change in the temperature of the experimental device.

The $T\cdot \ln(t/\tau_0)$ scaling can done by a nonsubjective algorithm.

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REFERENCES


