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RESEARCH MEMORANDUM

RESOLUTION OF ANNEALING EXPERIMENTS FOR THE STUDY OF NONEQUILIBRIUM STATES

By Philip Schwed

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NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS

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SUMMARY

Insight into the condition of a solid which is not in the equilibrium state may be obtained by determining the manner in which the properties of the solid change as it is annealed. In general, annealing is done either by subjecting the sample to a series of fixed temperatures or by steadily raising the temperature. In either case the way in which the property studied changes during annealing makes it possible to distinguish nonequilibrium states of sufficiently different activation energies. A resolving power is defined herein for each of these two techniques. Equations are derived which can be used to evaluate the merits of an annealing experiment on the basis of these definitions.

For annealing with a steadily rising temperature, the defined resolving power is nearly independent of the rate of temperature rise. The numerical results depend, of course, on the chosen definitions; but, because of the way they are related, they provide a valid basis for a comparison of these two (standard) methods. The separation in activation energies required for resolution increases as the order of the annealing process increases; it is somewhat greater for annealing with steadily rising temperatures than for annealing at fixed temperatures. In both cases, the necessary separation is found to be of the order of 10 percent of the smaller activation energy.

INTRODUCTION

The problem of matter in nonequilibrium states is of great importance in the case of solids, inasmuch as most solids encountered in practice are not in thermodynamic equilibrium. The departure from equilibrium manifests itself by the deviation of certain properties, such as stored energy, electric resistance, and so forth, from their equilibrium values; these properties, which allow conclusions to be drawn concerning the nonequilibrium states, are called index properties.

A better insight into the nature of these states is provided by the technique of annealing. In an annealing experiment, the rate at which

the material goes from the nonequilibrium state to some state closer to equilibrium under the influence of thermal agitation is determined by measuring the rate of change of one or several index properties as a function of time and temperature. If some of the atoms of the material are initially in the equilibrium state and the rest are initially in some single nonequilibrium state, the data obtained in such an experiment may be interpreted in terms of the number of atoms in either state and the energy barrier separating the two states. In the more usual case, however, a whole set of nonequilibrium states is available to the atoms of the solid, in which case the experiment does not give clear-cut information if the barriers separating the various nonequilibrium states from the equilibrium state are only slightly different in height. Thus, there is a definite restriction on the extent to which different states can be distinguished in such an experiment. 2249

Related to this limitation of the resolution is the question of how an annealing experiment is to be best performed. There are, in particular, two simple procedures available. An index property may be measured as a function of time at each of a series of fixed temperatures, each run being made at a higher temperature than the preceding; or the sample may be subjected to a steadily rising temperature (continuous annealing) and the index property measured as a function of time (and temperature). Each of these techniques obviously has its advantages and disadvantages so far as the experimental procedures involved are concerned. The present analysis was conducted at the NACA Lewis laboratory to determine the relative merits of these two techniques.

SYMBOLS

The following symbols are used in this report:

C_i y_i/n_i

E_i activation energy for passage from nonequilibrium state of ith type to equilibrium state, electron volts

δE difference in activation energies for two states which can just be resolved

n_i number of atoms in nonequilibrium state of ith type at time t

$n_i(0)$ value of n_i at $t = 0$

R ratio of initial values of $\left|\frac{dy_i}{dt}\right|$ for two states of equal initial y_i which are defined as just resolved in annealing at fixed temperatures

r time rate of temperature rise in continuous annealing

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T product of Boltzmann's constant and absolute temperature, electron volts

T_{ci} value of $T_{\rho i}$ for $\rho = 10^{-2}$ per second

$T_{\rho i}$ value of T for which $\left|\frac{dy_i}{dt}\right| \Big/ |y_i|$ is equal to ρ at a fixed temperature

T_{mi} value of T for which $\left|\frac{dy_i}{dt}\right|$ is a maximum in continuous annealing

δT_m difference in values of T_m for two states which are just resolved

T_0 value of T at $t = 0$

$T_{\frac{1}{2},i}$ value of T for which $\left|\frac{dy_i}{dt}\right|$ reaches one-half its maximum value

t time, seconds

y total deviation of index property from equilibrium value at time t

y_i value of deviation of index property from equilibrium value at time t due to atoms in i^{th} type of nonequilibrium state

$y_i(0)$ value of y_i at $t = 0$

γ order of reaction by which atoms return to equilibrium state

Δ $E_i(1/T_{\frac{1}{2},i} - 1/T_{mi})$

θ variable of integration

ν constant in expression $\nu e^{-E_i/T}$ for decay parameter of annealing process

ρ arbitrary critical value of $\left|\frac{dy_i}{dt}\right| \Big/ |y_i|$

ANALYSIS

In order to define the resolving power, it is necessary to consider a hypothetical experiment with a solid in which two defects of different activation energies are equally responsible for the initial deviation of the index property from its equilibrium value; and the extent of overlapping in their contributions to the annealing curve must be calculated. This overlapping is a function of the difference in activation energies and, consequently, it is possible to determine the minimum separation in activation energies for which the overlapping is less than a specified amount. The ratio of this minimum separation in activation energies to the smaller one of the activation energies will hereinafter be called the resolving power. (This definition is analogous to that used for the resolving power of a telescope, which is taken as equal to the angle subtended by two objects that can just be distinguished as separate at the position of the telescope (see reference 1). The resolving power thus defined increases as the merit of the instrument decreases.) The degree of overlapping on which it is based is, of course, to a certain degree arbitrary, but will be chosen for each specific type of experiment in a way to satisfy practical considerations. 2249

The definitions of the permissible overlapping are necessarily different for both standard types of annealing experiment, but will be chosen in a way that permits comparison of the merits of these experiments.

The physical situation may be represented as follows: There are available to the atoms of the solid a set of nonequilibrium states that will be distinguished by the subscript i. The number of atoms in the ith state will be designated by n_i and the height of the energy barrier between this state and the equilibrium state, by E_i. The states are numbered so that $E_i < E_{i+1}$. Then, if all annealing processes are of the same order, at any instant the rate at which n_i changes is given by (reference 2)

$$\frac{dn_i}{dt} = - \nu n_i^{\gamma} \exp(-E_i/T) \tag{1}$$

where

t time

ν constant

T absolute temperature (expressed in same energy units as E_i)

γ order of process

There are cases where ν depends on the temperature (reference 3, p. 191); however, the strong dependence of the exponential function on T makes the result insensitive to a dependence of the sort $\nu \propto \frac{1}{T}$, so that for most cases ν can be considered a constant.

Let the deviation of the index property from its equilibrium value be designated by y. (Thus y may stand for stored energy or for increase in electric resistance.) The portion of this deviation resulting from the presence of n_i atoms in the ith state will be designated by y_i; and the change in the index property will be assumed to be a linear function of the number of atoms in nonequilibrium states, so that

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$$y = \Sigma\, y_i = \Sigma\, C_i n_i \tag{2}$$

where C_i is assumed constant. The equation for the variation of y_i is therefore given by

$$C_i^{\gamma-1} \frac{dy_i}{dt} = -\,\nu y_i^{\gamma} \exp(-E_i/T) \tag{3}$$

Method 1 - Continuous Annealing

In this technique, the temperature of the sample under study is raised at a steady rate so that

$$T = T_0 + rt \tag{4}$$

where T_0 is the temperature of the sample at the beginning of the experiment and r is the time rate of temperature increase. The value of r is assumed sufficiently small that an atom initially in equilibrium will also be in equilibrium at the ambient temperature throughout the entire course of the process. For reasons of mathematical convenience, the case of a first-order process will be considered separately from the higher order processes.

When $\gamma = 1$ (first order), equation (3) becomes

$$\frac{dy_i}{dT} = -\,\frac{\nu}{r}\, y_i \exp(-E_i/T) \tag{5}$$

The solution of equation (5) is

$$y_i = y_i(0) \exp\left[-\,\frac{\nu}{r} \int_{T_0}^{T} \exp(-E_i/\theta)\, d\theta\right] \tag{6}$$

where $y_i(0)$ is the value of y_i at the time $t = 0$. In representing the results of an experiment, however, it is convenient to use dy/dt (or what amounts to the same thing, dy/dT) rather than y because a plot of dy/dt exhibits distinct peaks (if the difference of the activation energies is large enough) whereas one of dy/dT is a monotonic function (fig. 1).

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As already noted, the resolving power will be found by determining the least difference between the two barriers E_1 and E_2, which is consistent with an overlapping of the peaks no greater than a specified amount. The degree of overlapping which is tolerable depends essentially on the experimental error encountered; a practical definition of the permissible overlapping is the following:

Consider $\left|\frac{dy_i}{dt}\right|$ for two defects, y_1 and y_2, making initially equal contributions to y, $y_1(0) = y_2(0)$. (See fig. 1.)

Let $T_{\frac{1}{2},1}$ be the temperature corresponding to the point on the descending part of the $\left|\frac{dy_1}{dt}\right|$ curve for which $\left|\frac{dy_1}{dt}\right|$ is one-half its maximum value. Let $T_{\frac{1}{2},2}$ be the temperature corresponding to the point on the ascending part of the $\left|\frac{dy_2}{dt}\right|$ curve for which $\left|\frac{dy_2}{dt}\right|$ is one-half its maximum value. The curves $\left|\frac{dy_1}{dt}\right|$ and $\left|\frac{dy_2}{dt}\right|$ will be considered just resolved if $T_{\frac{1}{2},1} = T_{\frac{1}{2},2}$. Inasmuch as $\left|\frac{dy_1}{dt}\right|$ and $\left|\frac{dy_2}{dt}\right|$ are nearly equal at this temperature, the value of $\left|\frac{dy_1}{dt}\right| + \left|\frac{dy_2}{dt}\right|$ at this point is nearly equal to the maximum value of $\left|\frac{dy_1}{dt}\right|$.

It is first necessary, then, to find the value of T for which $\left|\frac{dy_i}{dt}\right|$ is a maximum, as has been done independently in reference 4. (That such a maximum exists is obvious on physical grounds.) This temperature T_{mi} is given by the solution of the equation

$$E_i\, T_{mi}^{-2} = \frac{\nu}{r} \exp(-E_i/T_{mi}) \tag{8}$$

Equation (8) follows by setting dy_i^2/dT^2 equal to zero, using expression (5) for dy_i/dT. For values of the constants ordinarily encountered, equation (8) may be solved by iteration.

If all energies and temperatures are in electron volts and times are in seconds, a reasonable set of values for the parameters encountered in an experiment involving a solid (reference 2) is

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$$\nu = 10^{13}/(\text{sec})$$

$$r = 10^{-5}(\text{ev/sec})(\text{approximately } 0.1^{\circ}\ \text{C/sec})$$

$$E_i = 1(\text{ev})$$

Equation (8) may be rewritten

$$T_{mi} = \frac{E_i}{\ln \nu - \ln r - \ln E_i + \ln T_{mi}^{\ 2}} \tag{9}$$

or after the specific values for ν, r, and E_i are introduced,

$$T_{mi} = \frac{1}{2.3\ (13 + 5 + 2 \log T_{mi})} \tag{9a}$$

Solving equation (9a) by iteration gives the value of T_{mi} as 2.95×10^{-2} electron volts. Approximately, then, for values of ν and r close to those given, equation (9) becomes

$$T_{mi} = \frac{E_i}{2.3 \log (10^{-3} \frac{\nu}{r})} \tag{10}$$

In particular, for values of ν greater than 10^8 per second, changing the rate r by a factor of 10 either way changes T_{mi} by no more than 10 percent, so that the temperature at which a state of particular energy anneals out is not strongly dependent on the way in which the experiment is performed.

The next step is to determine the temperature at which $\left|\frac{dy_i}{dt}\right|$ reaches one-half its maximum value. If this temperature is designated by the symbol $T_{\frac{1}{2},i}$, it follows from equations (5) and (6) that $T_{\frac{1}{2},i}$ is determined by

$$\ln 2 = E_i\left(\frac{1}{T_{\frac{1}{2},i}} - \frac{1}{T_{mi}}\right) - \frac{\nu}{r}\int_{T_{mi}}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\, d\theta$$

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When the change in variable $B = E_i\left(\frac{1}{\theta} - \frac{1}{T_{mi}}\right)$ is made, this expression becomes

$$\ln 2 = \Delta - \frac{\nu T_{mi}^{\,2}}{(rE_i)} \exp(-E_i/T_{mi}) \int_0^{\Delta} \left(1 + \frac{BT_{mi}}{E_i}\right)^{-2} e^{-B}\, dB$$

$$= \Delta - \int_0^{\Delta} \left(1 + \frac{BT_{mi}}{E_i}\right)^{-2} e^{-B}\, dB \tag{11}$$

where Δ is defined as $E_i\left(\frac{1}{T_{\frac{1}{2},i}} - \frac{1}{T_{mi}}\right)$ and use has been made of equation (8) in reaching the last result. If Δ is small compared with E_i/T_{mi}, the denominator of the integrand in equation (11) may be approximated by 1, and equation (11) may be replaced by the approximation

$$\ln 2 = \Delta - \int_0^{\Delta} e^{-B}\, dB = \Delta - (1 - e^{-\Delta}) \tag{12}$$

or

$$\Delta + e^{-\Delta} = 1.693$$

There are two values of Δ satisfying equation (11), namely,

$$\left.\begin{aligned} \Delta_1 &= -0.98 \\ &\text{and} \\ \Delta_2 &= 1.47 \end{aligned}\right\} \tag{13}$$

The error made by approximating $\left(1 + \frac{BT_{mi}}{E_i}\right)^{-2}$ by 1 is of the order of T_{mi}/E_i, that is, about 0.03 according to equation (10), thus justifying the approximation.

Only the problem of calculating the resolving power now remains. Each level may be specified either by E_i or by T_{mi}; the relation between these according to equation (10) is approximately

$$\ln T_{mi} = \ln E_i + \text{constant} \tag{14a}$$

so that

$$\frac{\delta T_m}{T_{m1}} = \frac{\delta E}{E_1} \tag{14b}$$

The percentage difference between the E's is thus equal to the percentage difference between the Tm's. However, by equations (13) and the definition of Δ,

$$\frac{\delta T_m}{T_{m1}} = (|\Delta_1| + |\Delta_2|) \frac{T_{m1}}{E_1} = 2.5 \frac{T_{m1}}{E_1} = 7.5 \times 10^{-2} \tag{15}$$

Thus the resolving power of this method is about $7\frac{1}{2}$ percent for a first-order process.

The same procedure may be applied to cases where the process is not first order. The equation governing this situation is, from equations (1) and (4),

$$\frac{dn_i}{dt} = -\frac{\nu}{r} n_i^{\gamma} \exp(-E_i/T) \tag{5a}$$

(The quantity n is used instead of y in order to avoid complicating equation (5a) and subsequent equations with unessential factors $C_i^{\gamma-1}$.) The integral of this equation is

$$(1-\gamma)^{-1}\left[n_i^{1-\gamma} - n_i^{1-\gamma}(0)\right] = -\frac{\nu}{r}\int_{T_0}^{T} \exp(-E_i/\theta)\, d\theta \tag{16}$$

and the condition that $\left|\frac{dn_i}{dT}\right|$ be a maximum is

$$\gamma = E_i\, T_{mi}^{-2}\, n_i^{1-\gamma} \frac{r}{\nu} \exp(E_i/T_{mi}) \tag{17}$$

which becomes, when equation (16) is used for $n^{1-\gamma}$,

$$\gamma = E_i\ T_{mi}^{-2}\left[n_i^{1-\gamma}(0) - (1-\gamma)\ \frac{\nu}{r}\int_{T_0}^{T_{mi}} d\theta\ \exp(-E_i/\theta)\right]\frac{r}{\nu}\ \exp(E_i/T_{mi}) \tag{18}$$

As was done in obtaining equation (11), the integral in equation (18) may be approximated by

$$\int_{T_0}^{T_{mi}} \exp(-E_i/\theta)d\theta = \frac{T_{mi}^{\ 2}}{E_i}\left[1 - \exp(-\Delta_{0,i})\right]\exp(-E_i/T_{mi}) = \frac{T_{mi}^{\ 2}}{E_i}\ \exp(-E_i/T_{mi}) \tag{19}$$

where $\Delta_{0,i}$ is $E_i(1/T_0 - 1/T_{mi})$; the last step follows if T_{mi} is sufficiently greater than T_0. (The error caused by neglecting $\exp(-\Delta_{0,i})$ is less than 10 percent for the values of the parameters assumed if $\frac{T_{mi} - T_0}{T_0}$ is greater than 10 percent; this condition represents no real restriction because otherwise appreciable annealing will occur at T_0.) The resulting equation for T_{mi} is

$$E_i\ T_{mi}^{-2}\ \frac{r}{\nu}\ n_i^{1-\gamma}(0)\ \exp(E_i/T_{mi}) = 1 \tag{8a}$$

For usual processes, $\nu n_i^{\gamma-1}(0)$ is of the order of 10^{13} per second (reference 3, pp. 242, 296), and the significance of equation (8a) is similar to that of equation (8).

In order to determine the resolving power, it is necessary to find the temperature $T_{\frac{1}{2},i}$ at which dn_i/dT attains one-half its maximum value. By using equations (5a) and (16), the following equation is obtained for $T_{\frac{1}{2},i}$:

$$\frac{1}{2} = \exp\left[-E_i(1/T_{mi} - 1/T_{\frac{1}{2},i})\right]\left[\frac{n_i^{1-\gamma}(0) - (1-\gamma)\ \frac{\nu}{r}\int_{T_0}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\ d\theta}{n_i^{1-\gamma}(0) - (1-\gamma)\ \frac{\nu}{r}\int_{T_0}^{T_{mi}} \exp(-E_i/\theta)\ d\theta}\right]^{\frac{\gamma}{1-\gamma}} \tag{20}$$

When the integral in the numerator is rewritten in the form

$$\int_{T_0}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\ d\theta = \int_{T_0}^{T_{mi}} \exp(-E_i/\theta)\ d\theta + \int_{T_{mi}}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\ d\theta$$

the quantity in brackets may be expressed as

$$1 - \frac{(1-\gamma)\ \frac{v}{r} \int_{T_{mi}}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\ d\theta}{n_i^{1-\gamma}(0) - (1-\gamma)\ \frac{v}{r} \int_{T_0}^{T_{mi}} \exp(-E_i/\theta)\ d\theta}$$

which may be further simplified to

$$1 - \frac{(1-\gamma)\ \frac{v}{r} \int_{T_{mi}}^{T_{\frac{1}{2},i}} \exp(-E_i/\theta)\ d\theta}{\frac{\gamma T_{mi} v^2}{E_i r} \exp(-E_i/T_{mi})}$$

by the use of equation (18). If the remaining integral is approximated as before, the following expression for Δ results:

$$\ln 2 = \Delta - \frac{\gamma}{\gamma-1} \ln \gamma + \frac{\gamma}{\gamma-1} \ln\left[1 + (\gamma-1)\ e^{-\Delta}\right] \tag{12a}$$

If $\gamma = 2$, this procedure leads to a net Δ (see equation (15)) of

$$\Delta = \left|\Delta_1\right| + \left|\Delta_2\right| = 1.75 + 1.74 = 3.5$$

The resolving power is therefore about 11 percent. For $\gamma = 3$, $\Delta = 6.5$ and the resolving power is about 20 percent.

Method 2 - Annealing at a Series of Fixed Temperatures

Corresponding to a given value ρ of the quantity $\left|\frac{dy_i}{dt}\right| \Big/ |y_i|$ there exists for a first-order process a temperature $T_{\rho i}$ given by

$$T_{\rho i} = \frac{E_i}{\ln \nu - \ln \rho} \tag{22}$$

It is practical to introduce in the following discussion a temperature T_{ci} "at which the ith state anneals" by choosing for ρ the reasonable value of 10^{-2} per second, so that for $\nu = 10^{13}$ per second

$$T_{ci} = \frac{E_i}{2.3\ (13 + 2)}$$

which is not very different from T_{mi} and rather insensitive to the special choice of ρ.

A definition for the resolving power must be established consistent with that used in the case of continuous annealing. A definition identical with that one is not applicable in the present case because the plot of dy/dt has a different shape: The curves of figures 1 and 2, which represent the situation qualitatively, show that in the case of continuous annealing dy_1/dt and dy_2/dt have peaks (fig. 1) which are absent in the case of annealing at a fixed temperature (fig. 2). In order to relate the definitions of resolving power for the two cases, the new definition of resolving power must be based on some relation between dy_1/dt and dy_2/dt which can be applied to both standard methods of annealing. In order to connect the two definitions, a system containing two states and "just resolved" (according to the old definition) in the continuous case will be defined as being also "just resolved" according to the new definition. The characteristic selected is the value of $\left|\frac{dy_1}{dt}\right| \Big/ \left|\frac{dy_2}{dt}\right|$ at the point for which $\left|\frac{dy_1}{dt}\right|$ is a maximum, assuming as before that $y_1 = y_2$ initially. For annealing at a fixed temperature, this characteristic is simply the value of $\left|\frac{dy_1}{dt}\right| \Big/ \left|\frac{dy_2}{dt}\right|$ at $t = 0$, whereas for the case of continuous annealing it is the value occurring at $T = T_{mi}$. This ratio for the case of continuous annealing is defined as R. The first step then is to determine R. From the derivation of equation (12), it follows that

$$\ln R = |\Delta_1| + |\Delta_2| - \left[1 - \exp(-|\Delta_1| - |\Delta_2|)\right] \tag{23}$$

which becomes, after introducing the values for Δ_1 and Δ_2 from equation (13),

$$\ln R = 1.68 \tag{24}$$

The value of $E_2 - E_1$ must now be determined such that the ratio $\left|\frac{dy_1}{dt}\right| \Big/ \left|\frac{dy_2}{dt}\right|$ takes on a value R for annealing at a fixed temperature. From equations (21), (22), and (24), it follows that this separation of energies is given by

$$E_2 - E_1 = 1.68\ T_{ci} \tag{25}$$

The corresponding resolving power according to the present definition is given by

$$\frac{E_2 - E_1}{E_1} = \frac{1.68}{\ln \nu - \ln(10^{-2})} \tag{26}$$

For $\nu = 10^{13}$ per second, equation (26) becomes $5.1\text{x}10^{-2}$.

If a similar calculation is made for the case of higher order processes with y_i replaced by n_i, as was done before (in equation (5a)), with assumptions about the relevant parameters similar to those stated previous to equation (12a), there results

$$\begin{aligned} \gamma &= 2 \qquad \ln R = 2.2 \quad \text{and} \quad \frac{E_2 - E_1}{E_1} = 6.5 \\ \gamma &= 3 \qquad \ln R = 4.3 \quad \text{and} \quad \frac{E_2 - E_1}{E_1} = 12.5 \end{aligned} \tag{27}$$

CONCLUDING REMARKS

Because the definitions of resolving power employed are consistent, the results obtained give a valid comparison of the two methods considered. The resolving power for annealing at a series of fixed temperatures has been shown to be about two-thirds of that obtained for annealing done with

a continuously rising temperature, so that on this basis, continuous annealing is an inferior technique. Furthermore, within the limits imposed by experimental difficulties in keeping the temperature of the sample uniform and in measuring the changes of index property, the resolution in continuous annealing is essentially independent of the rate of temperature rise.

The absolute values found for the resolving power are based on a specific definition. The relative values, however, provide a basis for the design of experiments. Thus, in annealing at a series of fixed temperatures, there is no gain in changing the absolute temperature in percentage by an amount less than the obtainable resolving power. Also, in interpreting an annealing experiment it is not proper to make assertions about the relative spacing of barriers smaller than the resolving power. For convenience, a table of the resolving power expressed in percentages is included.

Order of process, γ	Continuous annealing	Annealing at fixed temperature
1	7.5	5
2	11	7
3	20	13

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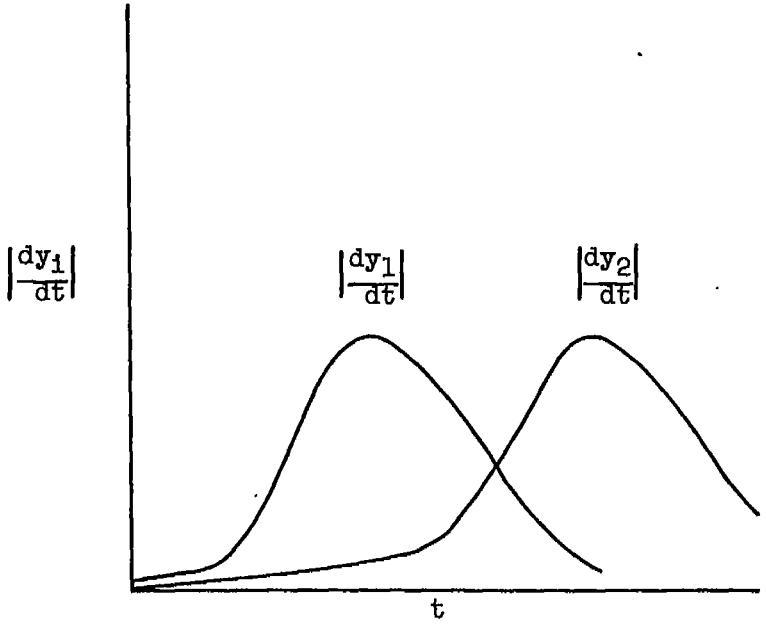

Figure 1. - Continuous annealing.

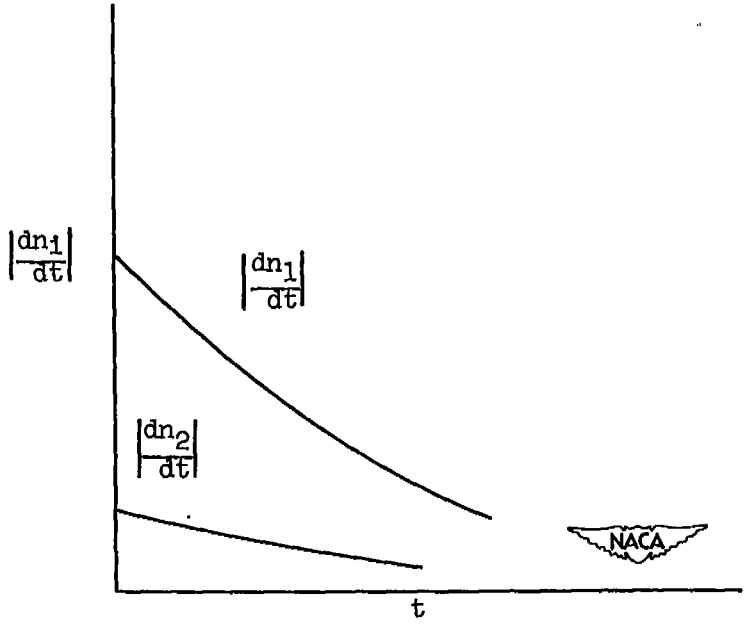

Figure 2. - Annealing at fixed temperature.

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