SHORT COMMUNICATIONS

EFFECT OF SLOW CYCLE COLD WORK FATIGUE ON THERMO-EMF OF NICKEL

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CHANGES in thermoelectric power (TEP) of pre-annealed nickel wire samples on low cycle cold work fatigue have been measured. The preannealing temperatures were 240, 290, 425, 600 and 750°C. Of these 290°C is the 'strain-energy-release' or 'recovery' temperature and 600°C is the 'recrystallization' temperature[1]. Wires annealed at two typical temperatures (290° and 750°C) show a decrease in TEP while the rest of the wires showed an increase in TEP on cold work fatigue. This interesting feature is reported in this note.

Nickel wire samples (0.25 mm dia.) were placed in glass/quartz capsules and sealed at 10^{-4} torr vacuum. These capsules were annealed in a muffle furnace at the above mentioned temperatures for 24 h. The wires were then electropolished, washed with methanol and stored in glass capsules. Their TEP was measured with respect to copper by a standardized ten wire potentiometer. The cold junction was dipped in ice-cooled mercury while the hot one was immersed in a mercury cup maintained at room temperature. Temperatures at the two

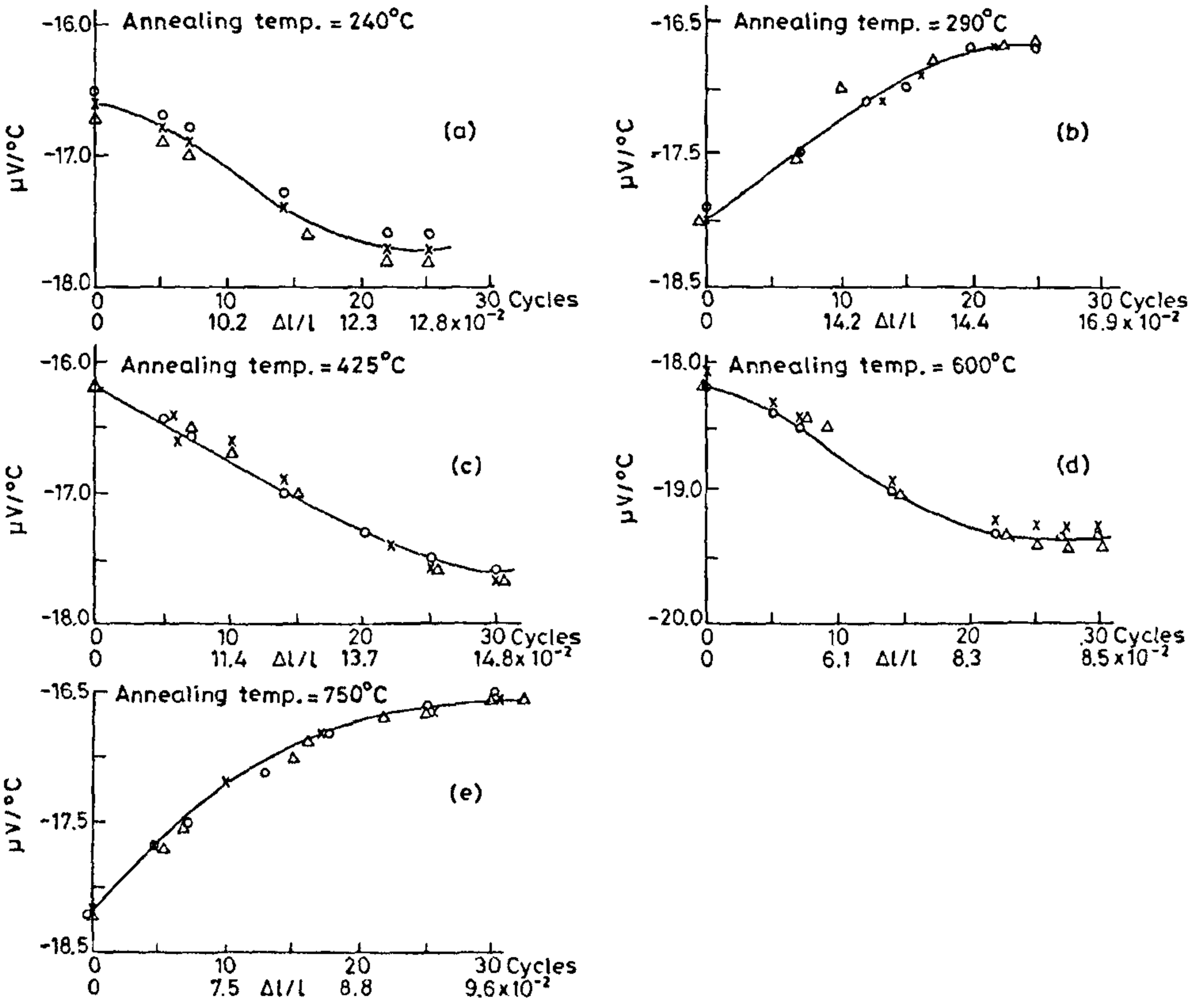

Figure 1. Variation of thermo-emf with the number of cold work cycles for differently annealed Ni wire samples.

junctions were read by mercury thermometers. Low cycle fatigue was effected[2] as described earlier[3]. The sample wire of known length (~1 M) was hung vertically under 1 kg wt in a rotatable iron rod of 0.78 cm dia. One operation of winding and unwinding constituted one cycle. After a few cycles the wire broke at the lower end. Immediately the elongation ΔL was measured. A small piece (~20 cm) was cut at the lower end and its TEP measured. The remaining wire was again subjected to a few cycles of operation and after breaking at the bottom the experiments were repeated.

Figure 1 shows the variation in TEP against the number of cycles each indicating $\Delta L/L$ observed which is a quantity proportional to the dislocation density. According to Brindley[4]

$$\text{TEP} \propto b \ln (x_0/x_n), \qquad (1)$$

where x_0 is the initial wire length and x_n the length after n cycles and b is a constant. From (1) it follows that on cold work, TEP should increase on the negative side. The TEP of nickel is $-17.3\,\mu$V/°C and becomes more negative. This explains the general trend of increase in TEP of Ni on cold-worked fatigue.

Now the question arises: why do wires annealed at 290°C and 750°C behave in the opposite manner? According to Friedel[5] at the recovery temperature (300°C in case of Ni) low angled boundaries are formed while at the recrystallization temperature (600°C for Ni) high angled boundaries are formed. Such boundaries act as sinks for dislocations which move on cold work from the interior of the grain towards the boundaries. Thus the total dislocation density decreases and so does TEP.

Figure 2 shows the recovery of TEP with time. According to Brindley the softening rate is

$$(-\mathrm{d}W/\mathrm{d}t) = \alpha W \exp(-Q/kT), \qquad (2)$$

Q is the activation energy needed to eliminate the cold work effect. No distinction is made for Q corresponding to different annealing temperatures. Figure 2 shows this to be essential. Samples pre-annealed at different temperatures show different amount of changes in TEP for the same number of cold work cycles. In the present case annealing was first done and then the cold work while in the case of Brindley the processes were in a reversed order.

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3. Nigam, A. N. and Gupta, A. C., *J. Electrochem. Soc. (India)*, 1984, **33**, 75.
4. Brindley, G. W., *Report on the strength of materials*, Bristol Conference of Materials, 1948.
5. Friedel, J., *Dislocations*, Pergamon Press, Oxford, 1964, p. 299.

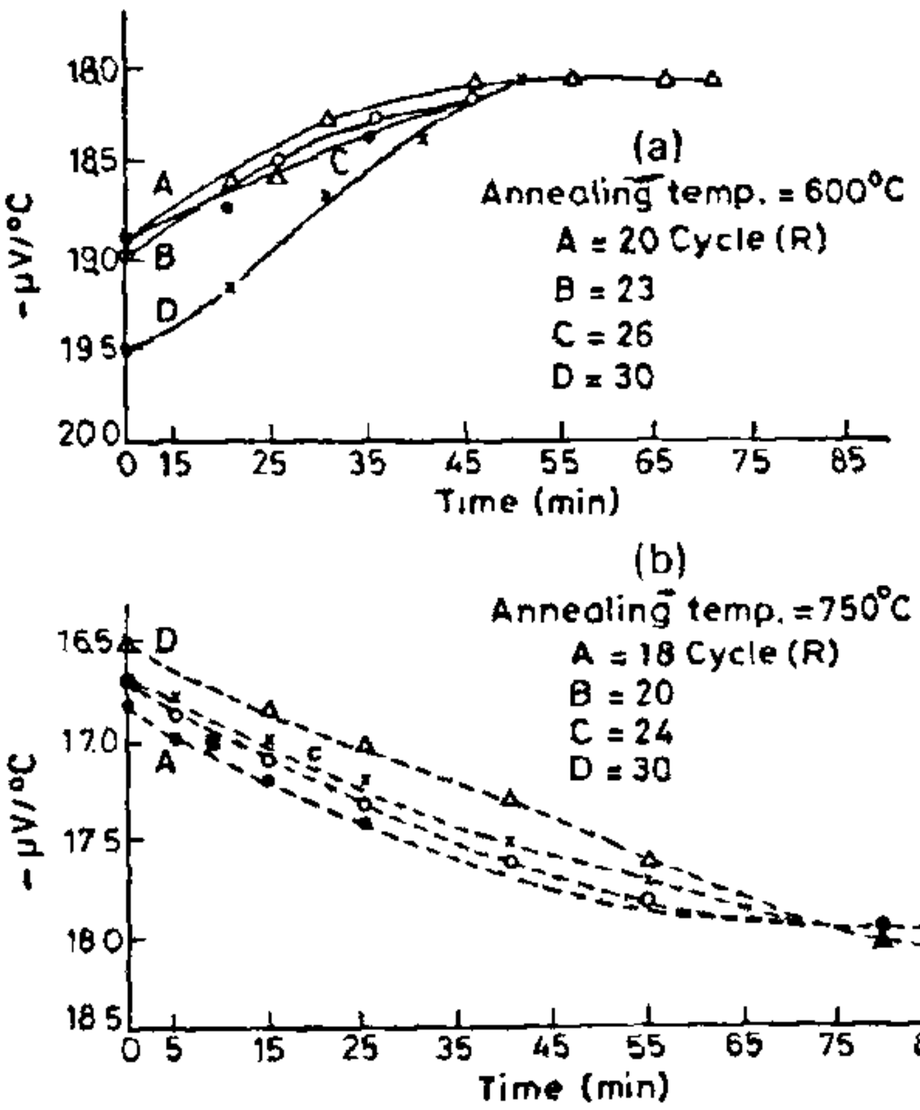

Figure 2. Recovery of thermo-emf in cold worked wires annealed at (**a**) 600°C, and (**b**) 750°C.

PROTOTROPISM IN THIOMORPHOLINE

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HETEROALICYCLIC compounds are familiar as pharmaceuticals[1] and biochemicals[2]. They are strongly basic[3] which is ascribed to their nonaromaticity. Piperidine, a six-membered saturated cyclic compound, contains a heteroatom and nitrogen. The presence of a second heteroatom may alter the basic properties and the compound may function as a dual donor[4]. Thiomorpholine, containing nitrogen and sulphur atoms is capable of donating non-bonded electrons available with both the heteroatoms[5]. It may also act as a dual proton acceptor. The prototropism of thiomorpholine is studied and its basicity is discussed here.