

## On the role of interface imperfections in thermoelectric nondestructive materials characterization

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This letter draws attention to a previously unnoticed artifact associated with the most common type of thermoelectric nondestructive materials characterization technique. It is shown that contact heating between the specimen to be tested and the reference electrode gives rise to a considerable offset in the measured thermoelectric voltage. The resulting bias significantly reduces the feasibility of thermoelectric measurements in nondestructive testing applications that require sensitive materials discrimination, for example, to sort metals of similar alloying content, to distinguish similar grades of heat treatment, and to detect slight variations in the thermoelectric power of metals due to hardening, texture, fatigue, etc. It is also suggested that the demonstrated intrinsic sensitivity of the thermoelectric contact technique to imperfect interfaces could be exploited for nondestructive detection of tightly compressed but metallurgically not bonded interfaces in spot welds, diffusion bonds, and other types of solid-state bonds. © 1998 American Institute of Physics. [S0003-6951(98)01830-0]

Ordinary thermocouples use the well-known Seebeck effect to measure the temperature at the junction of two different conductors. The electromotive force generated by the heat depends on the difference between the respective thermoelectric powers of the contacting metals and the junction temperature. Figure 1 shows the schematic diagram of the thermoelectric measurement as most often used in nondestructive materials characterization. The thermoelectric voltage is given by

$$V_{SR} = \int_{T_c}^{T_i} [S_S(T) - S_R(T)] dT, \quad (1)$$

where  $S_S$  and  $S_R$  denote the thermoelectric powers of the specimen to be tested and the reference electrode, respectively, and  $T$  is the temperature. Assuming that the relative thermoelectric power of the specimen with respect to the reference electrode  $S_{SR} = S_S - S_R$  does not change significantly between the temperatures of the hot  $T_i$  and cold  $T_c$  junctions, the thermoelectric voltage can be roughly approximated as  $V_{SR} \approx (T_i - T_c) S_{SR}$ . Ideally, regardless of the temperature difference between the junctions, only thermocouples made of different materials, i.e., materials of different thermoelectric power, will generate thermoelectric signal. The thermoelectric power of metals is sensitive to a variety of material properties that can affect the measurement. Clearly, chemical composition exerts the strongest effect on the thermoelectric properties and accordingly the basic application of thermoelectric materials characterization is metal sorting.<sup>1</sup> However, it is known that under special conditions materials of identical chemical composition can also produce an efficient thermocouple as a result of different heat treatments, hardening, texture, fatigue, etc., which can be further exploited for nondestructive testing of materials.<sup>2-4</sup>

Ideally, the thermoelectric power of the reference electrode should be somewhere halfway between those of the

extreme materials to be distinguished so that they produce thermoelectric signals of opposite signs. In an effort to apply this simple strategy to optimize a commercially available thermoelectric tester for texture and fatigue damage detection in titanium alloys, we recognized that a considerable thermoelectric voltage could be measured even when the reference electrodes were made from the very same material as the specimen to be tested. In the following we first present a few examples showing different degrees of thermoelectric offset and then investigate the physical reasons for this unexpected and previously unreported artifact which seriously limits the sensitivity of the thermoelectric technique to small variations in material properties.

Figure 2 shows examples of the thermoelectric signals measured at  $T_i \approx 300^\circ\text{C}$  between identical "specimen" and "reference" electrodes cut from the same thin wires of 1–1.5 mm in diameter for seven different materials. The schematic diagram of the experimental arrangement (with the reference electrode heated) is shown in Fig. 3. Both electrodes were of sufficient length to assure that the far ends that were

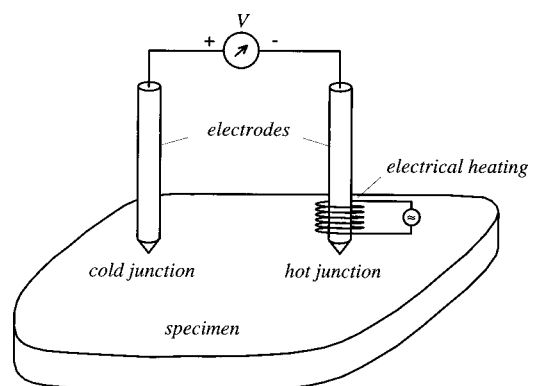


FIG. 1. Schematic diagram of the thermoelectric measurement as most often used in nondestructive materials characterization.

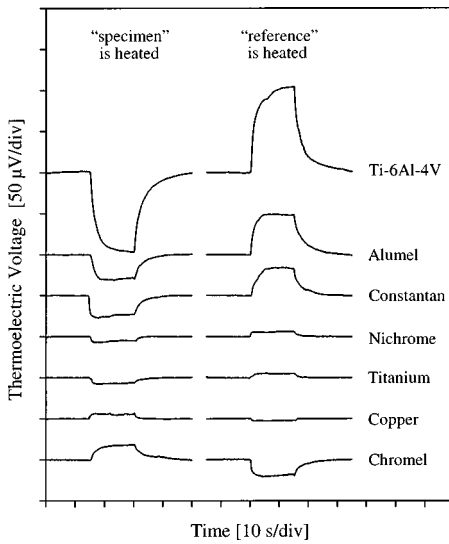


FIG. 2. Examples of thermoelectric signals from seven thermocouples formed by two identical metals compressed together ( $T_i \approx 300^\circ\text{C}$ ).

connected to a differential amplifier were cold, i.e., remained at room temperature. By definition, the wire connected to the noninverting input of the amplifier was considered to be the specimen and the one connected to the inverting input was the reference electrode. In spite of using identical materials for both electrodes, a significant thermoelectric signal was detectable in most cases. The measured thermoelectric signal was of basically the same amplitude but opposite sign for heating at the opposite sides of the junction. In comparison, when the junction was directly cooled by a FreezeIt<sup>®</sup> spray so that neither positive nor negative temperature gradient could occur through the interface no thermoelectric signal appeared between two identical wires. It should be mentioned that, like in essentially all thermoelectric nondestructive testing applications, the junction was formed by simply compressing rather than metallurgically bonding the electrodes. It was also noticed that the thermoelectric signal could be somewhat reduced but never completely eliminated by increasing the compressive pressure between the electrodes. Of course when the specimen and the reference electrode were parts of the same uncut wire, no thermoelectric signal was observed.

In order to understand the experimentally observed thermoelectric offset we must reconsider how the measured thermoelectric signal is produced as a result of the intrinsically coupled transport of electricity and heat via electron diffusion and phonon drag.<sup>5-7</sup> Let us consider the simple thermoelectric circuit previously shown in Fig. 3. The electrical potential difference  $V_{SR}$  between points  $S$  and  $R$  of constant ambient temperature  $T_S = T_R = T_c$  is

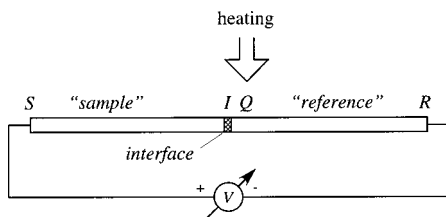


FIG. 3. The model used in analyzing the thermoelectric offset produced by an imperfect interface between the heated reference electrode and the specimen to be tested.

$$V_{SR} = \int_{T_c}^{T_i^{(-)}} S_S(T) dT + \int_{T_i^{(-)}}^{T_i^{(+)}} S_i(T) dT + \int_{T_i^{(+)}}^{T_i^{(-)}} S_R(T) dT + \int_{T_i^{(-)}}^{T_c} S_R(T) dT, \tag{2}$$

where  $T_i^{(-)}$  and  $T_i^{(+)}$  denote the temperature on the colder and warmer sides of the interface, respectively, and  $S_i$  is the unknown effective thermoelectric power of the imperfect interface. The third and fourth terms on the right side of Eq. (2) were separated this way to conveniently approximate the thermoelectric voltage as follows:

$$V_{SR} = \int_{T_c}^{T_i} [S_S(T) - S_R(T)] dT + [S_i(T_i) - S_R(T_i)] \Delta T_i, \tag{3}$$

where  $\Delta T_i = T_i^{(+)} - T_i^{(-)}$  denotes the temperature drop at the imperfect interface. Compared to the idealized form of Eq. (1), where the thermoelectric voltage is uniquely determined by the interface temperature alone, Eq. (3) also contains a correction term related to the temperature gradient at the interface. Without this correction term, the thermoelectric voltage  $V_{SR}$  is zero regardless of the shape and size of the specimen as long as it is homogeneous and isotropic, i.e.,  $S_S(T) = S_R(T)$ . Let us assume that the reference electrode is heated by a short thermal pulse at point  $Q$  as shown in Fig. 3 so that  $T_Q > T_S, T_R$  and consider the instantaneous thermoelectric voltage only so that neither  $T_S$  nor  $T_R$  has changed yet from their identical initial value of  $T_c$ . Clearly,  $V_{SR} = V_{SQ} - V_{RQ} = 0$  because  $V_{SQ}$  is identical to  $V_{RQ}$ . In reality, the thermoelectric voltage will not completely vanish because of the presence of the imperfect interface between the reference and specimen electrodes:

$$V_{SR} \approx [S_i(t_i) - S_R(T_i)] \Delta T_i. \tag{4}$$

An imperfect interface between contacting but metallurgically not bonded surfaces usually causes both reduced electrical and thermal conductivities, although the relative drop in the electrical conductivity tends to be larger than the corresponding drop in the thermal conductivity. Based on this general rule, the simplest physical approximation would be that  $S_i \ll S_R$ , and consequently the thermoelectric offset caused by the imperfect interface should be of opposite sign to the thermoelectric power of the reference material,  $V_{SR} \approx -S_R(T_i) \Delta T_i$ . At room temperature,  $S_R$  is negative for Ti-6Al-4V, alumel, and constantan but positive for chromel, nichrome, titanium, and copper. A comparison with Fig. 2 reveals that the measured thermoelectric offset was indeed of opposite sign to the thermoelectric power of the reference material in all the strongest cases. By far the strongest thermoelectric offset was exhibited by Ti-6Al-4V, the most popular titanium alloy used in the aerospace industry, that led us to our observation in the first place. The measured  $\pm 100 \mu\text{V}$  offset represents more than  $\pm 0.3 \mu\text{V}/^\circ\text{C}$  error in the thermoelectric power of Ti-6Al-4V, which is approximately  $-4.9 \mu\text{V}/^\circ\text{C}$  at room temperature.<sup>8</sup>

In the following we demonstrate that the above described thermoelectric voltage of the imperfect interface between the specimen and the reference electrode can render conventional thermoelectric material testers insensitive to

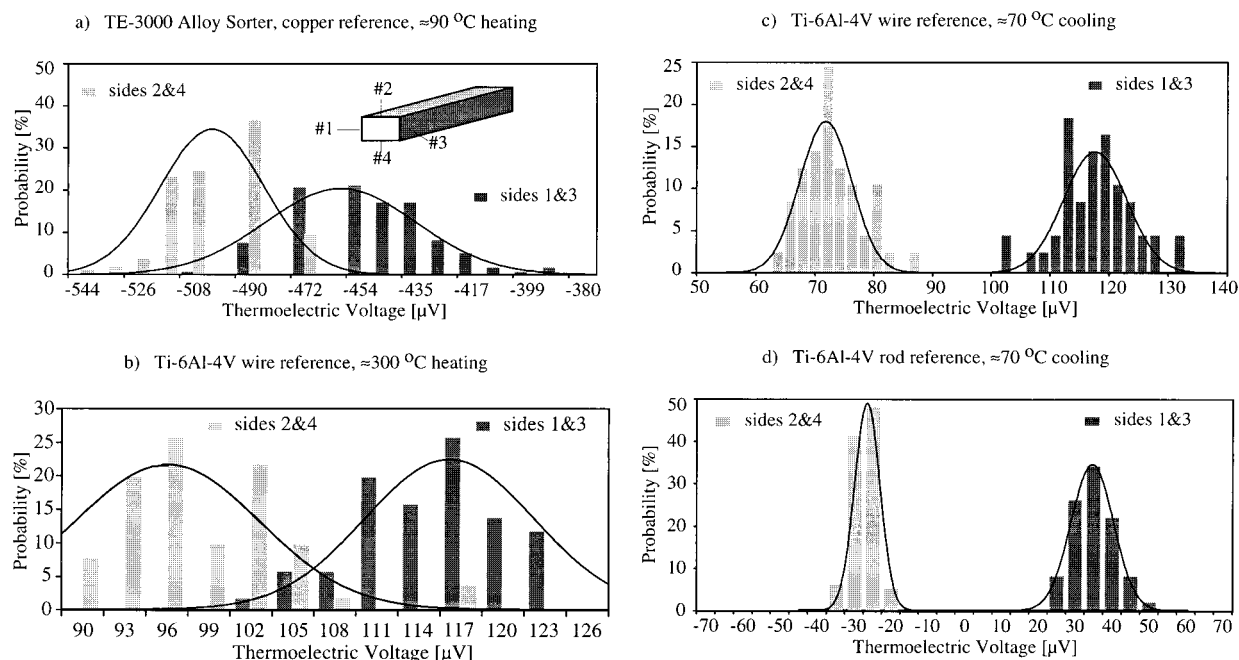


FIG. 4. Thermolectric texture assessment in Ti-6Al-4V by four different experimental procedures.

small material variations. Figure 4 shows the results of a series of experiments on thermolectric assessment of texture in Ti-6Al-4V alloy. This forged specimen exhibited a very strong anisotropic texture caused by the existence of a preferred crystallographic orientation in the polycrystalline structure that resulted in as much as 6% relative shear wave birefringence. In addition to this mechanical anisotropy, due to the dominantly hexagonal grain structure, the specimen also exhibited perceivable electrical conductivity anisotropy and was expected to show substantial thermolectric anisotropy as well.<sup>9</sup> First, a commercial TE-3000 Koslow alloy sorter was used, which has a copper alloy reference electrode ( $\approx +1.5 \mu\text{V}/^\circ\text{C}$ ) heated to approximately  $90^\circ\text{C}$  [Fig. 4(a)]. Since the reference electrode was very different from the titanium specimen to be tested, all sides registered at around  $-500 \mu\text{V}$  and sides (1 and 3) could not be distinguished from (2 and 4). Figure 4(b) shows the results obtained with a Ti-6Al-4V wire reference electrode heated to  $\approx 300^\circ\text{C}$ . This method slightly improved the separation between sides (1 and 3) and (2 and 4) with respect to the measurements using the copper reference, but far less than one would have expected based on the choice of the reference electrode. The main reason for the experimental uncertainty is the approximately  $+100 \mu\text{V} \pm 10\%$  offset caused by the imperfect interface, which was previously shown to exist between even two identical Ti-6Al-4V wires (see Fig. 2).

As we have mentioned above, the interface effect can be eliminated if the junctions between the specimen and the reference electrode are heated or cooled at the same time, which assures that there is no temperature gradient through the interface. Instead of heating, we used a commercial FreezIt<sup>®</sup> 2000 cooling spray to cool the interface region to approximately  $-70^\circ\text{C}$ . First, a Ti-6Al-4V wire was randomly chosen as a reference electrode [see Fig. 4(c)]. As expected, the large interface offset disappeared and we could separate sides (1 and 3) and (2 and 4). However, both sides gave positive readings, which means that the thermolectric

coefficient of the randomly chosen Ti-6Al-4V wire was beyond those of the two textured sides. After some searching, we have actually found a Ti-6Al-4V bar that separated the two sides ideally as it is shown in Fig. 4(d).

In conclusion, the thermolectric offset produced by an imperfect interface can substantially affect the nondestructive testing of certain materials. On the other hand, the demonstrated intrinsic sensitivity of the thermolectric technique to imperfect interfaces could be exploited for nondestructive evaluation of tightly compressed but metallurgically not bonded interfaces in spot welds, diffusion bonds, and other types of solid-state bonds and might lead to new inspection methods to detect distributed fatigue cracking in metals.

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