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Duerig, Albrecht, Gessinger

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T. W. Duerig, J. Albrecht, and G. H. Gessinger

SUMMARY

An alloy based on the Cu-Al-Ni ternary system has been developed at the research center of Brown, Boveri & Co., Baden, Switzerland, which provides a fully reversible (two-way) shape memory effect at significantly higher temperatures than those afforded by commercial memory alloys such as NiTi and Cu-Zn-Al. The higher temperature capability of this alloy could open new fields for the application of the shape memory effect, particularly in thermal switching and protection devices. After suitable deformation and processing, a shape change is observed while heating the alloy through the temperature interval from 175 to 190°C. This shape change can be completely reversed during subsequent cooling from 155 to 125°C. The magnitude of the reversible strain produced by this alloy is 1.5%; somewhat higher strains can be achieved if lower memory temperatures can be accepted, and conversely, better high temperature capabilities can be achieved by accepting smaller reversible strains. The memory effects in this alloy have been found to be unaffected by short overheatings to temperatures as high as 300°C.

INTRODUCTION

Since the discovery of shape memory some 40 years ago,[1] engineers and scientists both have been searching for cost-effective applications of the effect.[2-6] A large fraction of the more "successful" uses of shape memory have been "one-time" uses, in which the component is expected to perform a shape memory movement only once during its lifetime: couplings for joining thin-walled tubes, for example. If the memory effect is to be reused, the shape recovery obtained during heating must be fully reversible during subsequent cooling. Although such a "two-way" behavior can be produced in many memory alloys, applications which take advantage of this reversibility—thermal motors, control devices, thermal switches, etc.—have been limited, until now, by the low memory temperatures, the vulnerability to overheating, and the relatively poor thermal fatigue resistance of the available alloys. One example of such a reversible application is in thermal protection devices—devices designed to shut off a heat source whenever an overheating condition exists. Clearly a person's definition of "overheating" depends very much on the exact application intended, but generally speaking, this means temperatures well over 100°C—well beyond the capabilities of currently available alloys.

The purpose of this paper is to describe the development of a new shape-memory alloy offering higher memory temperatures than those currently available. The alloy is a copper-based alloy, containing 14.2 wt.% aluminum and 3.3 wt.% nickel. While earlier publications have described some of the more scientific characteristics of the alloy,[7-10] this publication contains a more general overview of the properties offered by this new alloy and the rather unique processing route used to achieve these properties. Before focusing on the behavior of Cu-Al-Ni itself, however, it is useful to review some of the more basic aspects of conventional shape-memory, and to explain in more detail why the properties offered by conventional alloys may not be particularly well suited for many potential shape-memory applications—particularly those requiring a reversible thermal switching action. After doing so, the Cu-Al-Ni alloy system will be introduced, along with a processing route which was developed to overcome many of the problems presented by more conventional alloys. Finally, there will be a more detailed discussion of the properties of Cu-Al-Ni, as well as of the stability of these properties during exposures to particularly harsh conditions: overheating, fatigue, and the like.

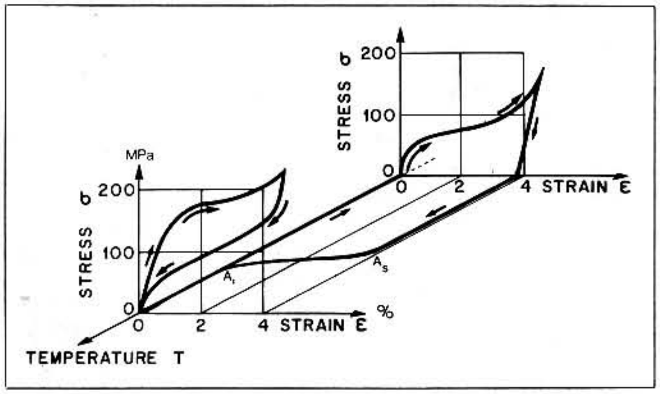

Figure 1. Tensile stress-strain-temperature map for NiTi shape-memory alloy.

CONVENTIONAL SHAPE MEMORY

The best way to introduce what can be described as the "classical" shape-memory effect, is to examine the behavior of NiTi—the first and, perhaps, the most widely recognized of the shape-memory alloys—during tensile deformation.

Two stress-strain curves are shown in Figure 1. The curve in the upper right of the figure corresponds to deformation below the alloy's M_F temperature, while the alloy is in a fully martensitic state. At a relatively low stress level, there is a stress plateau, during which strain can be accommodated, or stored, within the martensite structure (the details of this will be discussed below). When the load is released, one observes an elastic unloading, as would be observed in any other "normal" material.

By contrast, the stress-strain curve presented in the lower left corner of Figure 1 corresponds to a NiTi alloy deformed just above its A_F temperature, while the alloy is completely austenitic. In this case, the martensitic phase must be "stress induced" before the applied strain can be accommodated by the martensite structure; thus the same strain plateau exists, but at a higher stress level. More importantly, when the applied load is removed, the martensitic structure is no longer stable, and reverts through a shear mechanism to the austenitic phase. During this reversion, any strain which was being stored by the martensite is lost, and the material reverts to its original, pre-deformation, geometry. This is called a "pseudo-elastic" effect, and can be of use in applications requiring very high elastic strains, such as in certain types of springs.

The third axis in Figure 1, temperature, illustrates the shape-memory effect itself. If the material is deformed in its martensitic state and unloaded, there is, as has already been pointed out, a permanent strain. If the material is heated at some later time to its A_F temperature, the martensite and associated strain disappears and the original shape is recovered.

Thus the term "shape memory" means that the material remembers its pre-deformation shape, and reverts to this shape upon heating.

What then happens to the material geometry during subsequent cooling? Martensite must form if the material is cooled below its M_S temperature, but what may not be clear is whether it is the strained or the unstrained martensite which forms (i.e., the martensite structure and shape found prior to deformation, or that found after deformation). To understand this, one must examine exactly how the strain is being accommodated, or stored, in the material on a microscopic scale. In the as-quenched structure, the martensite plates are randomly oriented, and each individual plate is internally twinned in a random manner. When a load is applied, certain martensite variants and twin orientations are stabilized and begin to grow at the expense of others. Thus the material is able to store, or accommodate, strain by a combination of detwinning and reorientation mechanisms. Clearly there is a limit to the amount of strain that can be accommodated by such a process, and when this limit is approached, the slope of the stress-strain curve, $d\sigma/d\varepsilon$, must begin to increase, until, finally, true dislocation yielding is observed. If the amount of deformation found in a material is less than that required for dislocation yielding, all of the strain stored in the material is recovered during heating, and the randomly oriented and twinned martensite structure is again formed during cooling below M_F. Because there is a shape change during heating, but not during subsequent cooling, this is called a "one-way shape memory effect." Conversely, when the alloy is deformed by an amount sufficient to introduce or move dislocations, only part of the deformation can be returned during heating; the result is an imperfect one-way effect. In this case, however, a partial

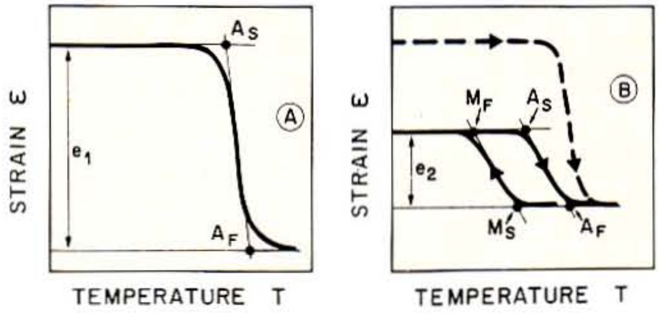

Figure 2. Illustration of a) the one-way shape-memory effect, and b) the two-way shape-memory effect. Note in b, that the A_S and A_F temperatures during initial heating are higher than those measured once full reversibility is achieved.

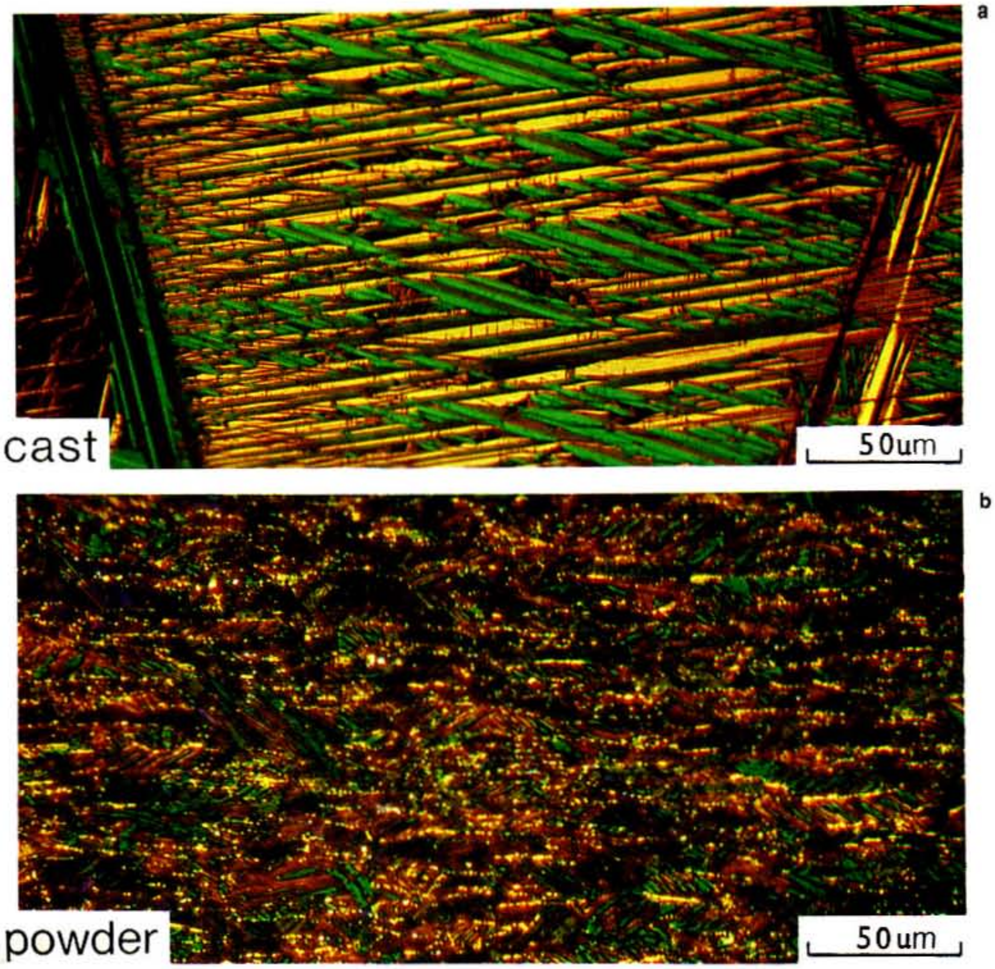

Figure 3. Comparison of cast and powder processed Cu-Al-Ni alloys. Both alloys are shown in the solution-treated-and-quenched condition.

return to the deformed geometry is noted during subsequent cooling through M_S. This means, that it is the detwinned and reoriented martensite that is formed, and not the original martensite structure; the presence of dislocations stabilizes the strained martensitic structure. This is generally termed a "two-way shape-memory effect," and is the effect which is of primary interest in this paper.

Both the one-way and the two-way effects are illustrated in Figure 2. The one-way effect, illustrated by the solid line in Figure 2a, is the most widely exploited form of shape memory, being used primarily as tube couplings and electrical connectors. The two-way effect is illustrated by the solid line in Figure 2b, and for reasons summarized in the introduction, is being exploited only on a limited laboratory scale for applications such as thermal switches, motors and actuators of various types. Also defined in Figure 2 are the magnitudes of the one-way and two-way effects themselves, denoted e_1 and e_2, and various temperatures key to defining a shape-memory movement, denoted M_S, M_F, A_S, and A_F.

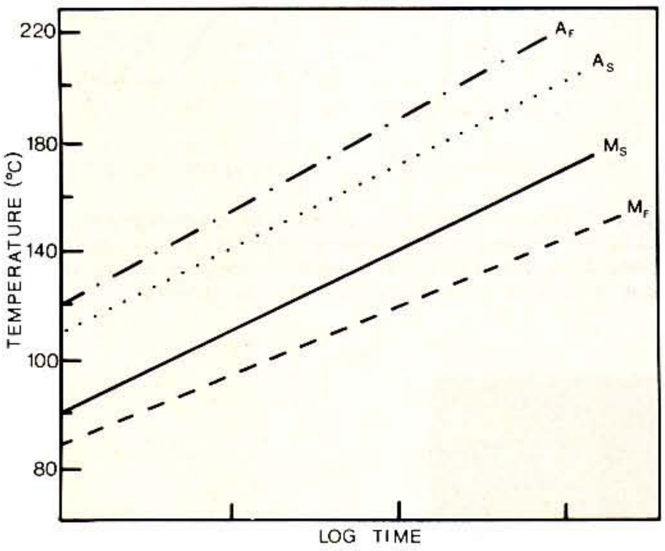

Figure 4. Schematic of the effect of aging on martensite stability.

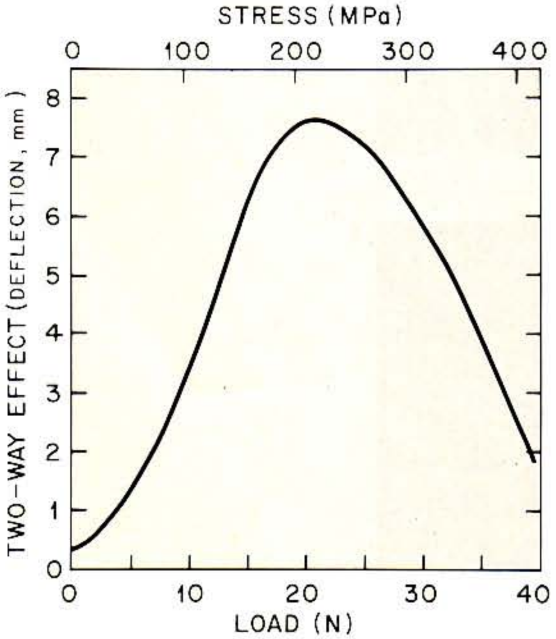

Figure 5. Illustration of how the two-way effect is affected by applying a load resisting the one-way effect.

As was mentioned in the introduction, one specific potential application for the two-way effect is in thermal protection devices, usually designed to open an electrical circuit whenever an overheating condition is sensed. A survey of such applications has shown that a switching temperature during heating (defined as $[A_S + A_F]/2$) above 160°C is required if shape memory metals are to seriously compete in this marketplace; the temperature at which the shape change is reversed $[M_S + M_F]/2$) is not as critical. Moreover, this temperature must be achieved while maintaining a reasonably large and distinct two-way movement, even after severe overheating and repeated use. Table I summarizes those properties we consider to be necessary in a successful candidate alloy for switching applications.

Table I: Targeted Shape-Memory Properties

Switching temperature: (during heating)	> 160°C
Two-way memory strain:	> 1.2 %
Switching interval: (temperature change required to produce 1.2% strain during heating)	< 40C°
Stability of memory effect:	
• Overheating to 300°C	> 60 minutes
• Thermal fatigue	> 1000 cycles
• Creep (30 MPa at 220°C)	> 7 days

THE DEVELOPMENT OF THE Cu-Al-Ni SHAPE-MEMORY ALLOY

Several alloys exhibiting shape memory effects (Cu-

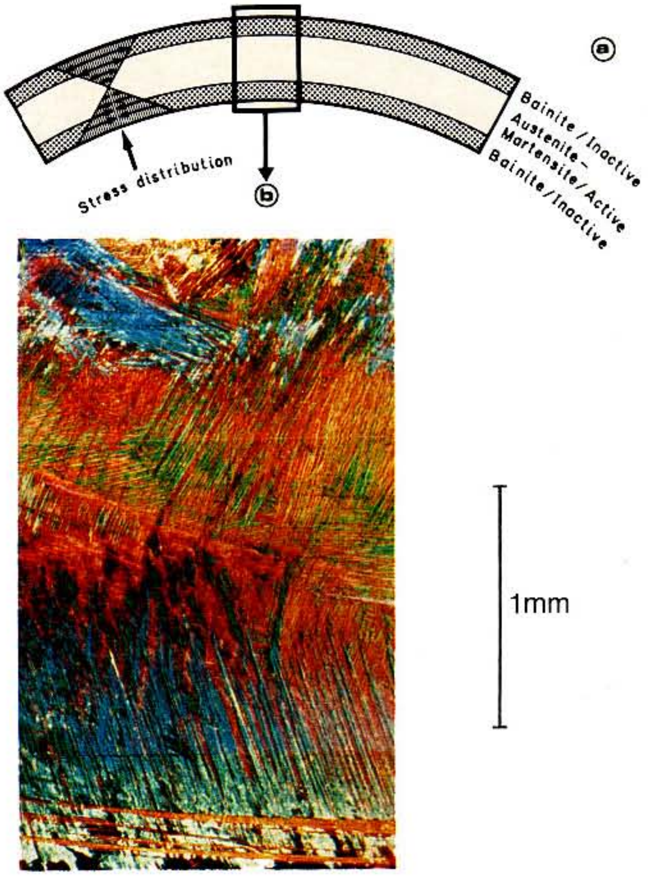

Figure 6. The effect of "shape stabilization" on bainite formation, shown a) schematically, and b) in an actual cast Cu-Al-Ni specimen.

Zn-Al, Cu-Al-Ni, and Ni-Ti-X ternary alloys) were initially investigated to find out which alloys offered the highest likelihoods that a sufficiently high switching temperature could be developed. Of these, the Cu-Al-Ni alloy system seemed most likely to be able to develop high memory temperatures at reasonable costs. After examining alloys with various aluminum and nickel contents, an alloy with 14.2 wt.% Al and 3.3 wt.% Ni was determined to produce the best combination of shape-memory movement and temperature. However, as will be seen, the properties of the alloy when processed via a normal route were still inadequate, and thus a rather complex processing route had to be developed before the desired properties were achieved. Before discussing the specific problems encountered and their solutions, it is necessary to describe some of the fundamental metallurgy of the Cu-Al-Ni system.

Table II: Comparison of Cast and Powder Properties of Cu-Al-Ni

	Cast Alloy	Powder Alloy
Grain Size (μm)	1500	20
UTS (MPa)	440	800
YS (MPa)	360	400
Ductility (%)	0.6	5-7

The Cu-Al-Ni system is metallurgically quite complex, particularly with compositions near that given above. The austenitic phase has the DO_3 structure, which is an ordered FCC superlattice structure with aluminum atoms at the face center and corner positions, and Cu and Ni atoms at the edge and interior positions. If the alloy is slowly cooled from the austenitic phase (stable above ~ 650°C), and $\alpha + \gamma_2$ eutectoid will form. If, on the other hand, the alloy is rapidly quenched through the M_S temperature, the parent DO_3 phase undergoes a martensitic transformation to either the γ' or the β_1' structure, depending on the exact composition of the alloy. X-ray tests have shown that both phases can be simultaneously present in the Cu-14.2Al-3.3Ni alloy described here.

As has been previously shown in single crystals,[8] and later in polycrystals,[11] Cu-Al-Ni undergoes further shear transformation to the α_1' and/or the β_1'' structures during straining. Although it would be out of place here to present the details of these transformations, it should be pointed out that these four martensitic phases (β_1', β_1'', γ', and α_1') are simply different stacking sequences of the same ordered basal plane; this means that to transform from one structure to another, only a rearrangement in the stacking sequence is involved. Electron microscopy investigations of the internal martensite structure have shown that the stacking composition of the martensite is extensively faulted, and, in fact, that any given plate can contain more than one type of stacking sequence (more than one martensite structure).

In addition to the austenitic, eutectoid, and martensitic phases, there appears to be an isothermal, intermediate phase similar to that reported in Cu-Zn-Al.[12] Moreover, it was found that the precipitation of this intermediate phase could be accelerated by the application of an external stress. This phase will henceforth be referred to as a bainitic phase, since it was found that it shares some of the properties of a martensitic phase (such as the influence of an applied stress upon its formation), and some properties of a diffusion controlled process. This is also consistent with the terminology set forth earlier with respect to Cu-Zn-Al.

With the above background, we can now examine the problems faced while attempting to develop the properties listed in Table I. Conventional processing (casting, hot working, solution treating, and quenching) was unsuccessful in developing these properties in three specific respects, i.e., inadequate ductility, insufficiently high memory temperatures, and inadequate two-way effect. The solutions to each of these problems will now be individually discussed.

Improving Ductility

Cu-Al-Ni alloys are inherently quite brittle. For example, when Cu-14.2Al-3.3Ni is cast, it has a room temperature tensile ductility of only 0.6%. Clearly if one requires a 1.2% two-way effect (Table I), one must be able to deform the material a great deal more than this. Examination of the fractured material showed that failure was entirely intergranular. Further, it was not possible to refine the grain size of the cast material; hot working would temporarily refine the grain size, but recrystallization and rapid grain growth was unavoidable during subsequent solution treatment. This problem was solved by manufacturing the alloy through powder metallurgical means. Three pre-alloyed powder compositions were blended, cold compacted, sintered, and hot swaged into rods. Powders and sintering technique were intentionally chosen to introduce oxide particles into the material. Such oxides are chemically inert (as far as the martensitic transformation is concerned), but serve to pin grain boundaries and thereby retard, or eliminate, the rapid grain growth found in the cast alloy. One should also note that the oxides are preferentially formed on the aluminum-rich powder particles, and thus the composition of the austenite matrix is not the overall composition of the conglomeration; there is a leaching of aluminium from the metal matrix to the oxides. Figure 3 shows a comparison of the cast and the powder metallurgy versions of the alloy. The white phase in Figure 3b is the oxide particles. The success of this refined grain size in enhancing ductility is shown in Table II. In addition to a tenfold increase in ductility, a sizable increase in strength is observed.

Extending the Temperature Range

After processing via the powder metallurgy route outlined above, an A_F temperature of 110°C was measured in the 14.2%Al + 3.3%Ni alloy; this temperature can be easily reduced by altering the Al or Ni content, but cannot be increased beyond 110°C without severely affecting the magnitude of the memory effect. Although this A_F is higher than that of Cu-Zn-Al and NiTi, it is still some 50C° below that shown in Table I. It was found, however, that the stability of the martensitic phase(s) could be increased by isothermally aging the austenitic phase in the 250-400°C range; an increase in martensite stability means, of course, that it forms and reverts at higher temperatures. As is illustrated in Figure 4, all four parameters, A_S, A_F, M_S, and M_F, increase during aging.

Enhancement of the Two-Way Effect

Although the above treatment was successful in producing an alloy with the appropriate memory temperatures, the material exhibited only a negligible reverse shape memory effect. To understand why, one needs only compare the stress-strain behavior of Cu-Al-Ni with that of NiTi. While the slope of a NiTi stress-strain curve flattens out initially then becomes steeper again (Figure 1), Cu-Al-Ni fractures while the stress-strain slope is still positive. When the strain exceeds that which can be absorbed by the martensitic structure of NiTi, dislocation motion begins and deformation continues; Cu-Al-Ni, on the other hand fractures before the onset of dislocation motion. Whereas in NiTi, it is this non-recoverable strain due to dislocation motion which gives rise to the reverse effect, all the strain is recoverable in Cu-Al-Ni. The one-way effect is always perfect, and consequently there is essentially no two-way effect.

How can the one-way effect in Cu-Al-Ni be hindered, or be made imperfect, and a two-way effect introduced? Or expressed in another manner; how can one force the reoriented, or deformed, martensitic structure to form during cooling, instead of the original, randomly oriented and faulted marensite? One solution is to "stress-direct" the austenite-to-martensite transformation. If the cooling is done in conjunction with an externally applied stress, and if the stress is in the same direction as the original deformation, then the same martensite structures will be formed as were formed during the original deformation, and thus the deformation strain will be produced. To demonstrate this concept, strips of Cu-Al-Ni were bent around a semicircular die. After deformation, one end of the strips was held fast in a rigid mounting, while the other end was left free, with a thin wire attached. The wire was then drawn over a pully, to a counterweight. The angular position of the pully, and thus the position of the free end of the shape-memory strip, was measured by means of an angular transducer. The element was then immerged into an oil bath, which could be heated and cooled in a controlled manner. In this manner the deflection of the strip could be continuously measured as a function of temperature and counterforce. The results of such an experiment are shown in Figure 5. As the stress in the direction of the original deformation was increased, so was the two-way effect. Note that the counterweight works against the one-way effect, and thus inhibits it. At very high loads, the one-way effect is inhibited to such a large degree that it becomes limiting to the two-way effect (the reverse effect can never be larger than the forward effect). Thus there is an ideal load for artificially inducing a two-way effect.

This concept of using a counterforce to artificially induce a two-way effect is, in many applications, practical. One could, in principle, design a device so that the shape-memory actuator acts against a conventional spring. There are many applications, in fact, where a constant load working against the one-way shape-memory movement is inherently present, such as in thermal motor or pumping applications, for example. To develop an alloy usable for a wide range of switching applications, however, one cannot assume that a constant force working against the desired heating movement is available; one must develop a two-way effect within the material, not rely upon the outside assistance of component designers.

In the case of a bending element, or any other shape giving rise to deformation inhomogeneities, there is one rather interesting solution to the problem, incorporating the stress-induced bainitic phase described earlier. Figure 6a illustrates the stress distribution through the thickness of a strip subjected to a bending stress. Stresses are maximum at either face, and there is a neutral axis in the middle. If one were to age a strip subjected to such as stress, one would expect an acceleration of the bainitic reaction at the two faces, where the stresses are maximum. The bainitic reaction is a diffusionally controlled process, and thus one would expect, and, in fact, one observes, a deterioration of the shape-memory process in bainitically transformed microstructures. Consequently, one can create a strip with three distinct regions—two inactive zones, each on either side of a central, bainite-free, active zone which still exhibits a shape-memory effect. Figure 6b shows a cross-section of a bent and aged strip. The white plate-like phase at the two surfaces is the bainite (verified by hot stage microscopy), and the brightly colored plates are martensite.

The bainitic layers provide a mechanism for stress relaxation, and thereby establish a new equilibrium shape for the material. During subsequent heating, the undisturbed core material tries to produce a perfect one-way shape memory effect, but is hindered by these inactive surface layers, effectively acting as two springs opposing the one-way movement. During cooling, these "springs" provide a force similar to that of the counterweight in Figure 5, and thereby induce a two-way effect. Because this treatment has the effect of stabilizing the deformed shape, we have chosen to call it a "shape stabilization" treatment. Clearly the thickness of the two inactive zones in comparison with the active core is critical in maximizing the two-way effect, and the aging conditions required to achieve the optimum relationship are complex, depending upon both component geometry and the magnitude of the original deformation.

PROPERTIES OF THE MEMORY ALLOY

To properly characterize the properties of Cu-Al-Ni, one must first describe both the conventional, bulk properties, and the shape-memory characteristics of the alloy, and then describe the stability of these properties during elevated temperature exposure, repeated cycling, and creep.

Physical Properties and Shape Memory Characteristics

Some of the key mechanical and physical properties of the alloy itself are shown in Table III. Of these, the electrical resistivity is of particular importance in most shape-memory applications since the heating of the mem-

Table III: Properties of Selected Shape-Memory Alloys

	NiTi-X	Cu-14Al-3.3Ni	Cu-Zn-Al
Yield strength (MPa)	580	400	80 - 200
Ultimate tensile strength (MPa)	830	800	500 - 600
Ductility, %	54	5 - 7	15
Fatigue strength, MPa, 10^6 cycles	350	270	—
Specific heat, J/K·m^3	2.980 x 10^6	3.52 x 10^6	3.06 x 10^6
Thermal conductivity, J/K·m·s	10	75	120
Electrical conductivity, 1/Ω·m	2 x 10^6	9 x 10^6	14.2 x 10^6
A_s	-100...+70	175	-200...+100
A_F	—	191	—
M_s	—	156	—
M_F	—	123	—
One-way effect, %	8	7	4.5
Two-way effect, %	5	1.6	1
Minimum temperature interval to produce 1.2 % strain on heating	—	15	—

ory material is often done by direct electrical means, by designing the Cu-Al-Ni element as an integral part of an electrical power circuit.

Table III also shows some of the more important shape-memory characteristics of the alloy, averaged from over 100 bending elements manufactured according to the above described process. In thermal switching or protection applications, the most important characteristic is usually the temperature at which switching takes place during heating, taken to be an average between A_s and A_F. Although the mean value for this "switching temperature" (T_s) is 180°C, T_s can be lowered by changing the alloy composition slightly, or can be increased to as high as 200°C through small modifications in the processing route (with some small sacrifice in the magnitude of the two-way effect). The two-way effect of 1.6% is far in excess of the values shown in Table I, as is the "steepness" of the switching characteristics: $e_2/(A_F - A_S)$.

Property Stability

The next issue which must be addressed concerns the stability of the alloy when exposed to particularly harsh operating conditions. In order to define stability, one must first determine at what point the material ceases to perform its intended duty, and then define this as a "failure." Clearly if a structural material were to break, it would represent a failure. In the case of shape-memory alloys, however, there are many other ways an element can fail, based, for the most part, on the loss of, or changes in, the shape-memory characteristics of the material. For example, if the switching temperature were to change during service, this could represent a failure. In our case, it was estimated that only a 10% increase or decrease in switching temperature could be tolerated before the element was defined to have failed. Similarly, a 30% relative loss in the magnitude of the two-way effect could be tolerated. Also if the element were to change its shape during service by more than 7% due to stress relaxation, it would be considered to have failed. With these criteria in mind, we can now look at the ability of the material to survive temperature overheats, thermal cycling, and creep environments.

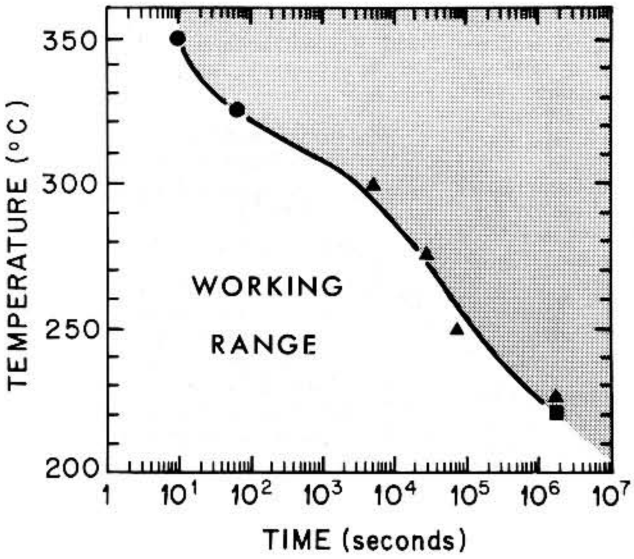

Figure 7. The effect of overheating on the shape-memory properties of Cu-Al-Ni. The envelope curve itself represents length of time a test element was able to survive at temperature (without a superimposed load). The individual symbols indicate which memory parameter gave rise to failure: circles indicate an overall shape change, triangles indicate a migration in memory temperature, and squares indicate a loss in two-way effect.

In most thermal protection applications, the temperature of a shape-memory switching element should increase only until its switching temperature is reached; at this point, the heat source is generally removed. In an ideal system, then, a shape-memory element should not be exposed to temperatures above its A_F temperature: there is a lag time, or delay between the removal of the heat source and the time at which the maximum temperature is reached. This delay is normally electrical in nature, and not due to a mechanical delay in the shape-memory effect itself. Thus one must look at the stability of the element during exposures to relatively high temperatures, well above the A_F temperature of the alloy. The results of tests conducted to measure the ability of Cu-Al-Ni to retain its shape-memory properties during high-temperature exposures are summarized in Figure 7. Only the most critical shape-memory parameter is shown, i.e., the first parameter to exceed the limits set forth above. The initial material goal, that the alloy survive 300°C temperatures for 1 hour, is easily exceeded.

The second stipulation set forth in Table I was that the material survive 1,000 thermal switching cycles without failure, again using the criteria described above to define failure. The deterioration of all three failure criteria are plotted as a function of the number of cycles in Figure 8. The conclusion is that an element can survive at least 7,000 cycles (between 50° and 200°C) without failure. Although this may not be sufficient for thermal motors and the like, it is certainly sufficient for overload protection applications.

Finally, there is a requirement that the material survive reasonable loads at high temperatures for extended time periods. Only one test was done to verify this. A bending specimen was loaded so that the maximum load anywhere on the specimen was 30 MPa, and then left at 220°C for two weeks with no detectable deterioration in the alloy's performance. Although the temperatures to which an element can be exposed might be quite a bit higher than this, it is extremely unlikely that these high-temperature excursions would be experienced while still under load, since there is generally very little delay in the time needed to remove the physical load from the element in comparison with the time needed to remove the heat source.

The above stability properties exceed those presented in Table I, which were based on a thermal protection type of application. For applications whose normal func-

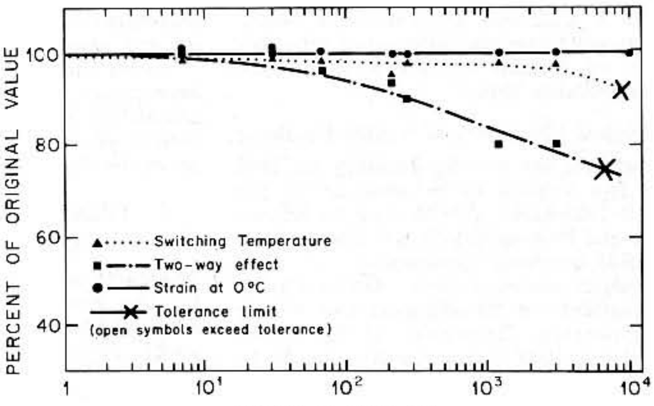

Figure 8. Loss of memory due to thermal fatigue in Cu-Al-Ni. The decays of three memory parameters are shown as a percentage of their original value and as a function of the number of cycles between 50°C and 200°C: drift in overall shape (measured at 50°C) migration (increase) in switching temperature, and magnitude of two-way effect. The bold X's illustrate the point at which a shape-memory property is estimated to have failed.

tion is to switch, such as thermal motor applications, the above stability goals may not be satisfactory. It would be possible, in such cases, to extend the fatigue life or the exposure survival time by adjusting alloy composition and heat treatment slightly. In such a case, it may be, however, that the other memory properties would suffer to a slight degree.

ACKNOWLEDGMENT

The work presented here should not be construed to be only the work of the authors. Since the original conception of the Cu-Al-Ni development program, several scientists have made major contributions to this project. O. Mercier was responsible for many of the advances in the Cu-Al-Ni development described here; K. Melton was responsible for much of the background and original shape-memory ground work; H. Riegger developed the powder processing route; and G. Schröder contributed to the organization and direction of the project. Special credit should be given to R. Sebalj and W. Weber for providing technical continuity and practical "know-how."

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