Forming Nitinol - A Challenge

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Abstract

Nickel-Titanium intermetallic compounds, commonly known as Nitinol alloys, exhibit the phenomena of thermal shape memory as well as superelasticity. The forming of parts from Nitinol requires a good understanding of the underlying mechanisms. This paper explains the thermo-elastic martensitic transformation and describes thermo-mechanical procedures to optimize the properties. The peculiarities of forming parts from Nitinol are highlighted.

1 Introduction

Nitinol alloys are widely used for applications like tube and pipe couplings, fasteners, actuators, and as of recently mainly for components for surgical and interventional instruments, as well as, because of their excellent corrosion resistance and biocompatibility, for implants /1/.

Nitinol alloys, equi- or near equiatomic compunds of Nickel and Titanium, are most commonly known for their superelasticity and thermal shape memory. These effects were discovered at the Naval Ordnance Laboratory in the USA in the late fifties and the alloys exhibiting the effects were called Nitinol. Nitinaol alloys will, after an apparent plastic deformation, return to their original shape when heated. The same materials, in a certain temperature range, can be strained up to approx. 8% and still will return to their original shape when unloaded. Both effects depend on the occurrence of a specific type of phase change known as thermoelastic, martensitic transformation. Shape memory and superelastic alloys respond to temperature changes and mechanical stresses in non-conventional and highly amazing ways. They are, therefore, sometimes called "smart materials".

On the other hand, Nitinol alloys can respond in strange ways to processing. During cold rolling, for example, some of the reduction in thickness is lost when the sheet is heat treated after rolling. Looking at the stress-strain curves of superelastic Nitinol and stainless steel, one can expect some issues with forming of Nitinol parts. A profound

understanding of the underlying mechanisms of shape memory is essential for the development of technically and economically efficient forming processes. In the following, we will describe the basics of the shape memory effects and discuss manufacturing processes for components made from Nitinol alloys.
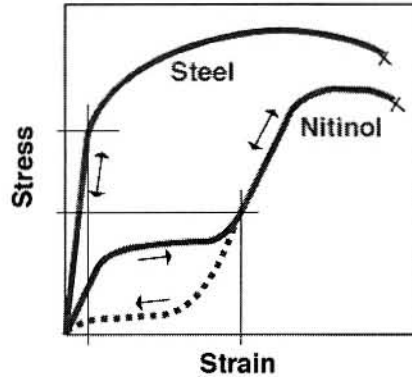

Fig. 1 Tensile Behavior of Nitinol and Stainless Steel

2   The Thermo-Elastic, Martensitic Transformation

Nickel-Titanium alloys with compositions around 50 at.% Ni and 50 at.% Ti undergo a solid state phase change on heating and cooling, the temparature of which is dependent on the exact alloy composition. The high temperature or parent phase is called austenite. It's crystal structure is B2 or Caesiumchloride. The low temperature phase, martensite, is monoclininc and heavily twinned. It can be easily deformed up to 8% strain by detwinning. The transformation from austenite to martensite and the reverse transformation from martensite to austenite are diffusionless, i.e. the atoms do not leave their positions in the lattice. Both transformations not take place at the same temperature .
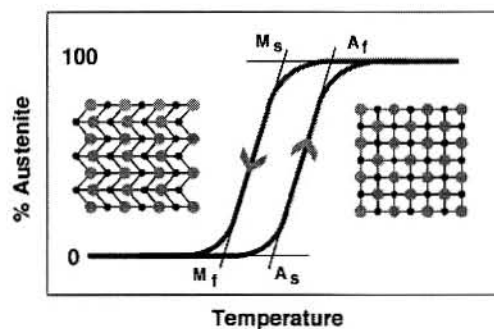

Fig. 2 Hysteresis and Transformation Temperatures

A plot of the volume fraction of martensite as a function of temperature provides a curve of the type shown schematically in Figure 2 together with the corresponding atomic arrangements. The complete transformation cycle is characterized by the following temperatures: austenite start temperature (As), austenite finish temperature (Af),

martensite start temperature (Ms) and martensite finish temperature (Mf). All temperatures are controlled by the composition and can be adjusted between ca −100·°C and +100°C /2/.

2.1 Shape Memory and Superelasticity

The mechanism of thermal shape memory is shown schematically in Fig. 3 /3/. Without external stress applied, there is no macroscopic shape change associated with the transformation from Austenite to Martensite. The transformation occurs without diffusion, i.e. the atoms remain on their lattice positions. In the martensitic condition (below Mf) the material can be easily deformed by detwinning, i.e. through a flipping-over type of mechanism until all twins have disappeared. This process occurs without the movement of dislocations. The maximum deformation that can be achieved by this process is 8%, i.e. a wire of one meter length can be "plastically" strained 8 cm. As long as the temperature remains below the transformation temperature, the wire stays deformed. Heating above the transformation temperature will cause the material to transform back into Austenite and the deformation will be recovered, i.e. the wire will go back to its original length of 1 meter.
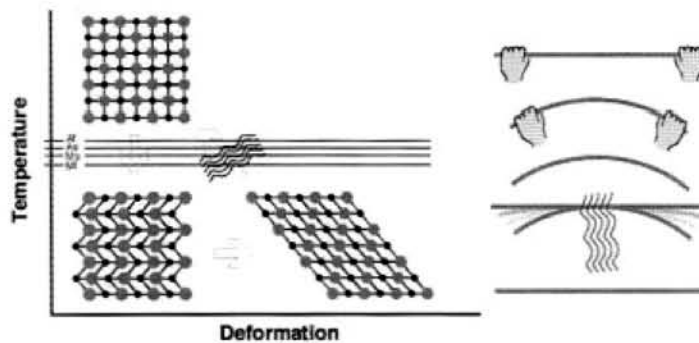

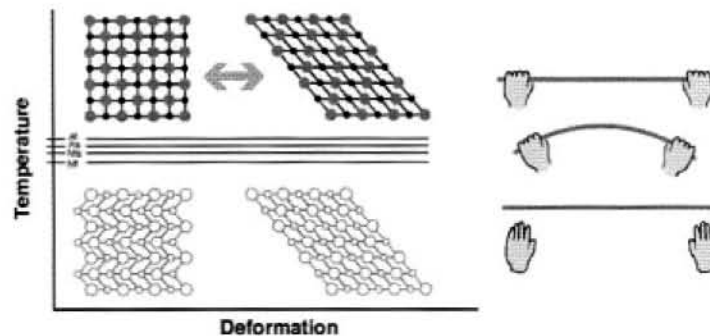Fig.3 Martensitic Transformation and Shape Memory Effect

Fig. 4 Stress Induced Martensitic Transformation and Superelasticity

If a stress is applied to a shape memory alloy in the temperature range between Af and a maximum temperature Md, martensite can be stress-induced. Less energy is needed to stress-induce and deform martensite than to deform the austenite by conventional mechanisms. Up to 10% strain can be accommodated by this process (single crystals of specific alloys can show as much as 25% pseudoelastic strain in certain directions). As austenite is the thermodynamically stable phase at this temperature under no-load conditions, the material springs back into its original shape when the stress is no longer applied. This extraordinary elasticity is also called pseudoelasticity or transformational superelasticity.

The design of shape memory components, e.g. fasteners or actuators, is based on the distinctly different stress/strain curves of the martensite and austenite, and their temperature dependence. Figure 5 shows tensile curves of a Ni-Ti alloy in the martensitic and austenitic conditions. While the austenitic curve (T>Md) looks like that of a "normal" material, the martensitic one (T<Mf) is quite unusual. On exceeding a first yield point, several percent strain can be accumulated with only little stress increase. After that, stress increases rapidly with further deformation. The deformation in the "plateau region" can be recovered thermally. Deformation exceeding a second yield point cannot be recovered. The material is then plastically deformed in a conventional way through dislocation movement.
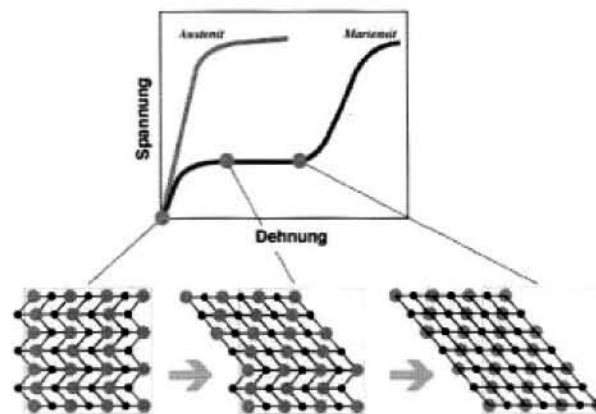

Fig. 5 Martensite Deformation by De-Twinning

If a component is deformed within the range of the plateau to, e.g. 6%, and then unloaded, it will stay in the deformed condition except for a small amount of elastic spring-back. The deformation can be recovered by heating to above the transformation temperature. Deformations beyond the plateau, or more specifically beyond the true yield point, cannot be recovered thermally. In this case the recoverability of the plateau deformation is reduced also.
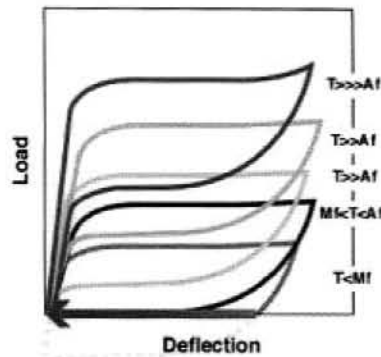
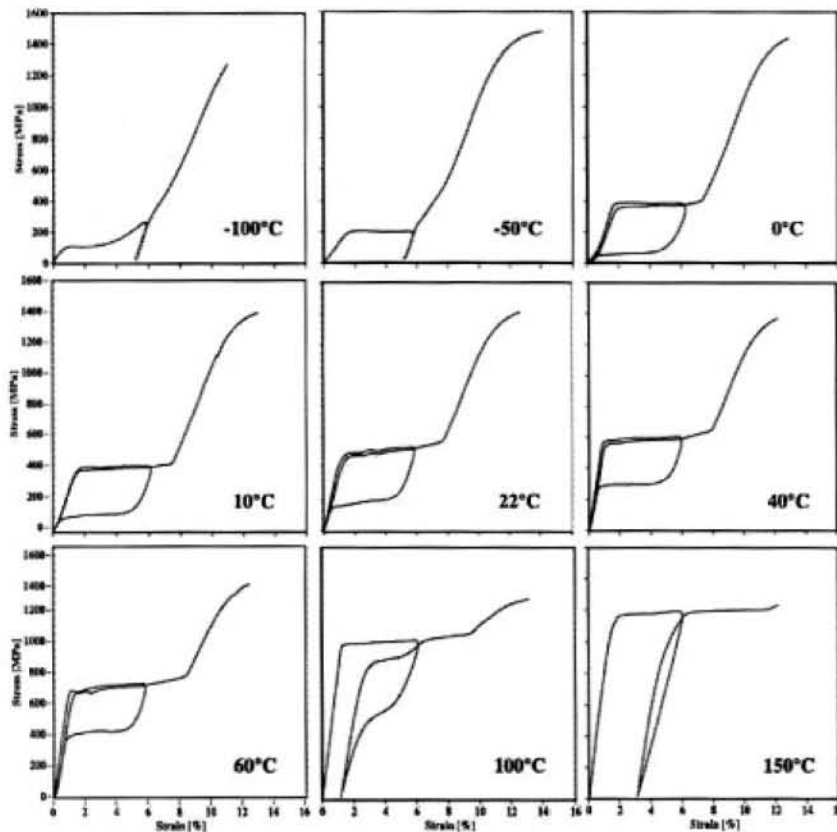Fig. 6 Temperatur Dependence of the Stress Hysteresis

Fig. 7 tensile Curves of a Superelastic Alloy at Various Temperatures

Fig. 6 shows qualitatively the temperature dependence of the tensile curves up to approx. 6% strain of a Nitinol alloy. Below Af, the material is "plastically deformed" (but thermally recoverable upon heating). Between Mf and Af the deformation will be recovered partially elastically on unloading. The other part can be recovered thermally. Above Af deformation occurs completely by stress-inducing and detwinning martensite. This deformation will be recovered elastically upon unloading. Increasing the temperature increases the plateu stresses and moves the entire hysteresis loop up. At the same time a non-revoverable portion appears with increasing temperature. At Md the

plateaus disappear completely, i.e. martensite can no longer be stress induced. The curve looks similar to those of normal materials. Fig. 7 shows a sequence of tensile curves at specific temperatures /4/.

The curves in Fig. 6 represent testing of a specific alloy at different temperatures. The same set of curves can be obtained by testing at room temperature (or a given test temperature) a group of alloys with different transformation temperatures with the curve with the lowest plateau stress belonging to the alloy with the highest Af and vise versa.

2.2   Optimization of the Properties

A combination of cold work and annealing is necessary to achieve optimum properties in Nitinol parts. This is particularly true for superelastic components and must be considered during the design of the part and during selection of the manufacturing process. As shown in Fig. 8, Nitinol wires in the as-drawn condition do not show the "flag shaped" stress/strain behavior. In order to achieve maximum superelasticity the material has to be heat treated at temperatures between 400 and 500°C. With this heat treatment the maximum achievable elastic strain and the shape of the hysteresis loop is controlled, as well as the plateau stresses and the non-recoverable strain /5/.
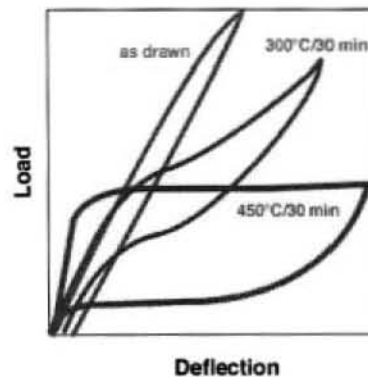

Deflection

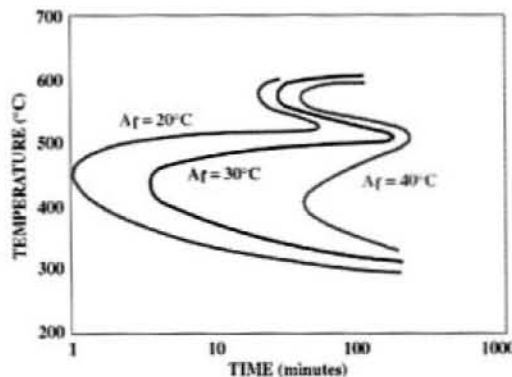Fig. 8 Tensile Behavior of a Ni-Ti Alloy in Different Conditions

Fig. 9 TTT Diagramm

Most importantly, however, in superelastic alloys this heat treatment controls the transformation temperature of the component through an aging process. In these alloys Nickel can be precipitated, a process that shifts the Ni:Ti ratio and thus the transformation temperature. Temperatures between approximately 0°C and 40°C can be achieved with the same alloy /5/. Fig. 9 shows a TTT diagram (temperature, time, transformation).

3   Manufacturing and Processing of Nitinol Alloys

3.1   Semi-Finished Materials

Melting of Nitinol alloys is typically done in vacuum by electron beam melting, arc melting, or induction melting. Of particular importance is the exact composition of Nickel and Titanium (a difference in composition of 0.1% causes a shift in transformation temperature of 10 degrees) as well as the amount of impurities from the atmosphere or the crucible material (mainly oxygen and carbon). The cast ingot is then processed by mostly conventional hot forming methods, like forging, swaging, rod and sheet rolling. While cold drawing of Nitinol wire and even tubing is relatively straightforward, cold rolling of sheets to thin dimensions poses some problems
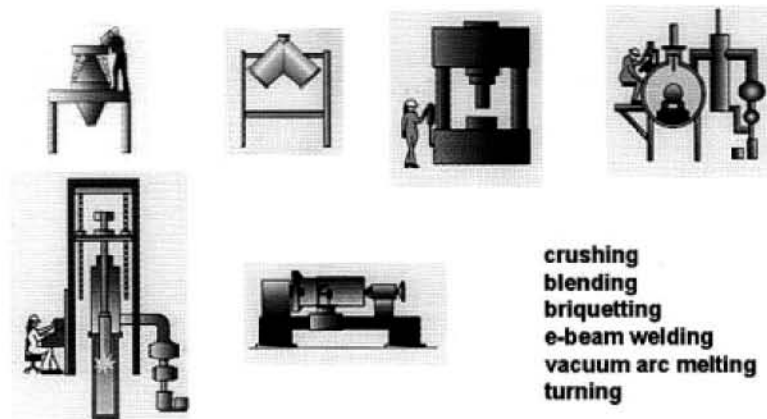

crushing
blending
briquetting
e-beam welding
vacuum arc melting
turning

Fig. 10 Melting of Ni-Ti Ingots

3.2   Processing of Nitinol Components

As described in previous chapters, the properties of superelastic and shape memory components are strongly dependent on the thermo-mechanical processing. Conventional forming techniques in most cases are not adequate because of the significant spring-back of the superelastic materials or the thermal recovery of the shape memory alloys. Machining techniques like drilling, milling etc. are possible, but tool wear is excessive.

In general, forming of Nitinol components is done by thermal shape setting using cold worked material. The parts are fixtured on shaping tools and heat treated at temperatures between 350 and 600°C in air or protective atmosphere.

In the following, we will describe some typical components and the methods to manufacture them.

3.2.1   Couplings and Fasteners

If a component after being deformed in the martensitic condition is constrained, i.e. physically prevented from returning into its original shape, then a stress is generated on heating /6/. Significant forces can be generated in this way. Tube and pipe couplings are among the oldest applications of the shape memory effect and have been produced for over 30 years. These sleeves are typically machined from solid barstock. Because of the poor material usage in machining sleeves (Nitinol chips cannot be reprocessed) attemps have been made early on to produce blanks by hot back-extrusion. It turned out, however, that this method was not cost effective, because of the excessive tool wear and significant re-work costs. Moreover, due to the different texture of the extruded material, shape memory properties were inferior compared with those of machined parts.
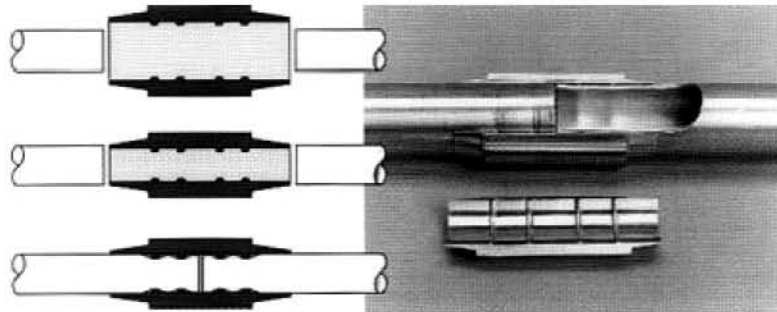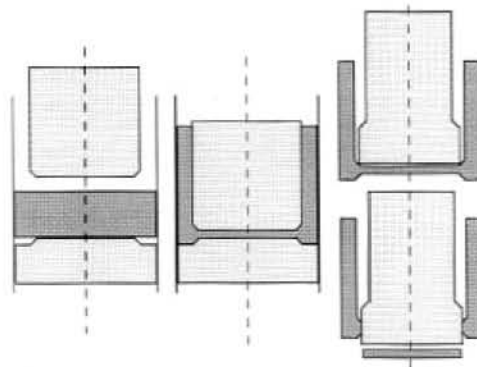

Fig. 11 Pipe Coupling

Fig. 12 Back-Extrusion of Coupling Blanks

3.2.2 Springs

Springs are preferred configurations for thermal actuators. They typically work against a reset mechanism like a steel spring. The Nitinol spring is designed such that at high temperatures the Nitinol spring is strong enough to compress the steel spring (in the case of compression springs). However, at low temperatures (in the martensitic condition), the steel spring is able to compress the Nitinol spring /7/.
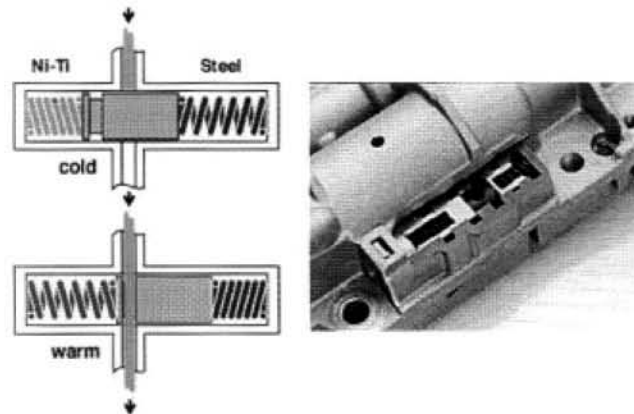

Fig. 13 Thermal Actuator in Pressure Control Valve of an Automatic Transmission /8/

Small quantities or complex spring geometries are typically produced by winding cold worked wire on a forming mandrel and heat treating in air or protective atmosphere. The spring will take the shape as fixtured. At the same time the transformation temperature can be adjusted to a certain degree. For volume production, custom made heated spring coiling machines are used. On these machines, the springs are produced with the final dimensions. In most cases, they require a post forming heat treatment for optimum properties.
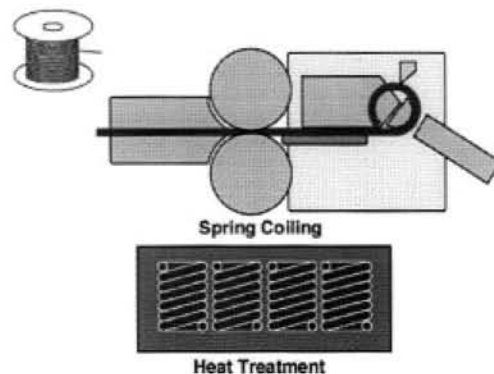

Fig. 14 Hot Spring Coiling

3.2.3 Superelastic Components

Superelasticity has found by far more applications than thermal shape memory. One reason for this might be the fact that control of the transformation temperature is not as

important as in the case of shape memory applications. As long as the transformation temperature, specifically Af, is below operating temperature the material will be superelastic. The medical industry has accepted Nitinol as a standard material in a multitude of applications /9/. However, even the consumer product industry uses Nitinol in increasing quantities. An excellent example is the superelastic eyeglass frame which was introduced about ten years ago and, in the meantime, is probably the most popular frame. Especially the temples are interesting from a manufacturing point of view, as they require fashion dictated non-uniform cross-sections along the length with varying superelastic properties. They should be superelastic, but allow adjustment in the ear region. The production sequence for a superelastic temple is shown in Fig. 15 and 16. A length of annealed wire is cold worked by rotary swaging to a reduced diameter. The undeformed end is hot coined. This section will be converted into a hinge component. A hole is drilled through the coined flat and the remaining end sheared off. To induce optimum superelasticity the part is heat treated. To make a section of the temple deformable, this section is fully annealed by conductive heating.
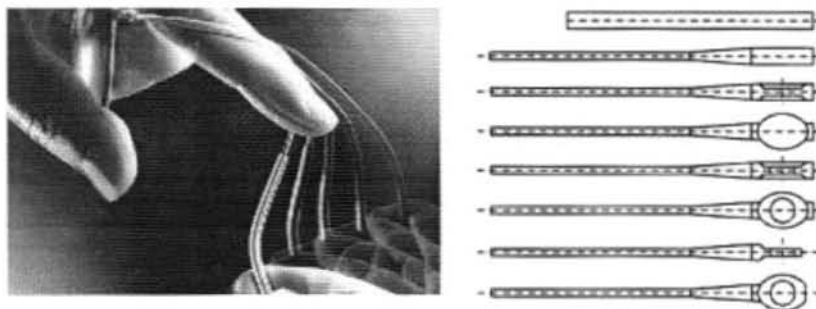

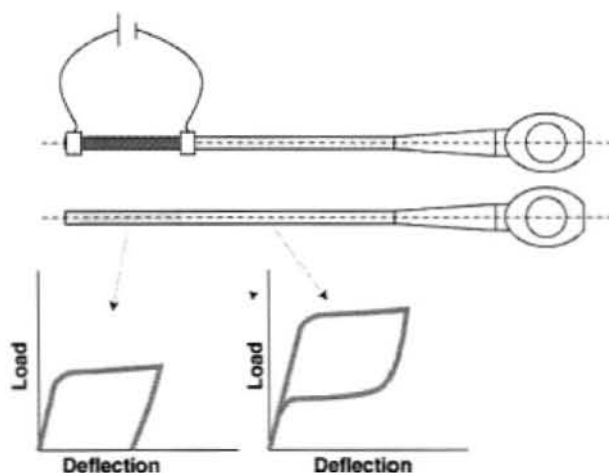Fig. 15 Manufacturing Sequence for Superelastic Eyeglass Temples

Fig. 16 Localized Annealing to Remove Superelasticy

The most important applications of superelastic Nitinol are medical applications. This sector has seen explosive growth during the past years and it continues to grow

significantly. In its simplest form a superelastic medical instrument is a formed wire, which can be straightened by passing it through a canula, catheter or other delivery device into the body. Inside the body at the treatment site, the delivery device is retracted and the wire deployed. It returns to its original shape elastically or through body heat. An early example is a tumor localizing hook used in mammography /10/. Production of these hooks is done by simply winding cold worked wire on a forming fixture and heat treating the arrangement. The hooks are then produced by cutting through the arrangement. This is shown schematically in Fig. 17.
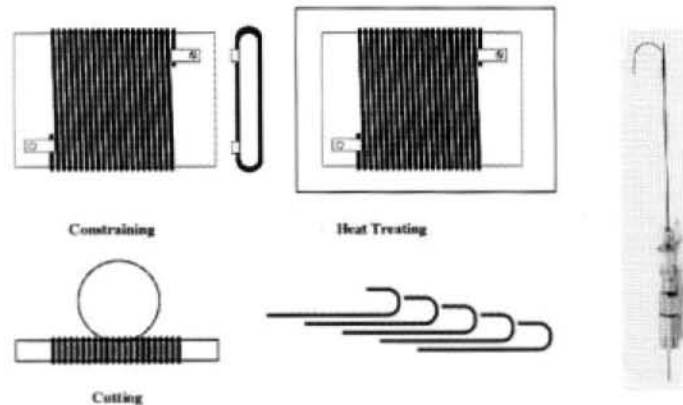

Fig. 17 Forming of Surgical Hooks

More complex instrument components can be formed by wire EDM from various starting shapes. Fig. 18 shows an interesting example of a hingeless grasper used in laparoscopic procedures. The functional part is a monolithic piece of superelastic Nitinol which is actuated by a sliding tube. While conventional, hinged graspers are difficult to disassemble and clean for sterilization because of the large number of individual miniature parts, the superelastic grasper is easily disassembled and cleaned. This part is fabricated by profil grinding a blank from a piece of cold worked rod. The contour of the jaws is cut by wire EDM. The jaws are forced open in a fixture and heat treated to set the open shape /11/.
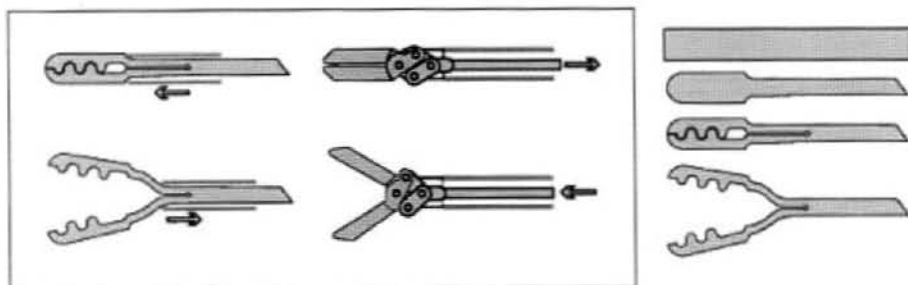

Fig. 18 Hingeless Grasper

Undoubtedly the most successful application of superelastic Nitinol is self-expanding stents /12/. Stents are vascular implants which help maintain a lumen in a diseased vessel or hollow organ. They are transported to the treatment site compressed inside a delivery system. After being released from the delivery system, the stents expand until they touch the vessel wall and further expansion is prevented. In general, stents are laser cut from Nitinol tubing with an expandable pattern. After cutting they are expanded by sliding on tapered mandrels and heat treated on the mandrels. Besides setting the shape of the stent, with this heat treatment the transformation temperature is typically set to approximately 30°C. Fig. 19 shows different sizes of as-cut stents (on a dime for size comparison) as well as a stent in the expanded condition (bent to show flexibility).
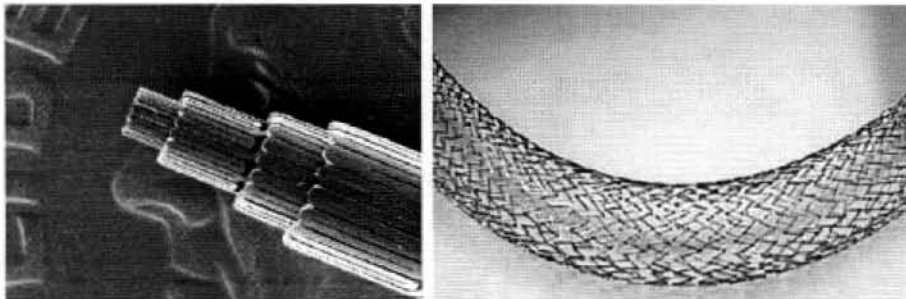

Fig. 19 Stents in the As-Cut and Expanded Condition

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