

Advancing Microsystem Technologies Through Electroplated Nanostructures

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Abstract

Factors affecting reliability and performance are the main concerns when dealing with microsystem components / devices with continuously shrinking external dimensions. In particular, microstructural non-uniformities leading to materials property variations across the microcomponent have presented major challenges. However, recent developments in the field of electroplated nanocrystalline materials can offer an avenue to address many of these technical difficulties.

In this paper, the deficiencies of metallic microsystem components currently in use are addressed, followed by a comparison which shows how nano-electroplated microcomponents can alleviate these problems. Materials properties enhancements that are achievable as a result of having nanostructured microcomponents will be discussed. The effects of grain size reduction on several specific performance indicators such as elastic energy storage capacity and wear resistance will be examined.

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Introduction

Although metallic components for microelectromechanical systems (MEMS) have been available since the 1980s, the microstructures of these microcomponents are still typically the same as for macrostructures produced by bulk processing. With the ever-decreasing size of the components and devices to less than $1000\mu\text{m}$, the tolerances for defects and non-uniformities in the materials and finished products are proportionally reduced. For example, the same defect density that would normally be acceptable in a gear 1cm in diameter can become detrimental in a MEMS gear having a diameter of only 1mm, thus greatly increasing the probability of catastrophic failures and significantly reducing the reliability of such a component. Today, most metallic MEMS components are fabricated using conventional electroplating techniques with little consideration regarding the structure-property relationships of the plated structure with respect to dimensional scaling¹.

LIGA (German acronym for Lithographic [lithography], Galvanoformung [electroforming] and Abformung [molding]) is the most widely used process to fabricate materials and components for MEMS, in which individual microcomponents are made by specialized electroplating techniques. Figure 1 shows schematically the various steps involved in a typical LIGA process.

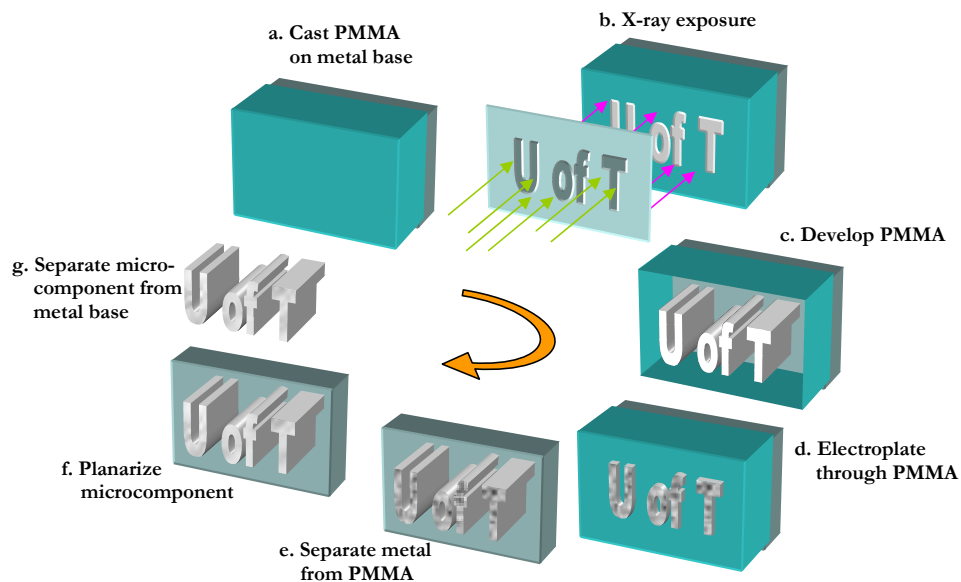


Figure 1 Schematic diagrams showing the various steps in a typical LIGA process; (a) PMMA cast on metal substrate, (b) X-ray exposure, (c) PMMA development, (d) electroplating through PMMA mold, (e) separating PMMA from plated component and substrate, (f) planarize microcomponent and (g) final product separates from substrate.

The process requires a polymer mold (e.g., PMMA) which is made by microlithography techniques (e.g., using synchrotron X-ray or UV irradiation). The developed mold then acts as a mask in the electroplating step, allowing only the exposed area(s) to be electroplated. The elec-

troplated structure, when removed from the mold is the finished MEMS microcomponent. The advantage of LIGA is that structures can be made of almost any material that can be electroplated at relatively low stress levels. These include nickel (Ni), copper (Co), gold (Au), silver (Ag), cobalt (Co) as well as nickel alloys (typically Ni-Co and Ni-Fe), with nickel being the most commonly LIGA-processed material ².

The development of LIGA processes for specific applications is not without challenges. Because the most important step in LIGA is based on electrodeposition, it is subjected to the same processing issues normally associated with electroplating practices. That is to say, the effect of processing parameters (such as bath composition and pH, operating temperature and current density, etc.) on the quality of the deposits directly translates to the degree of success of a specific LIGA process. However, hydrodynamic conditions in a mold cavity are quite different and much more difficult to control than for a conventional electroplating system.

In this paper, the microstructural deficiencies of currently available LIGA products are addressed, followed by an overview of what advantages nanocrystalline electrodeposits can offer to advance microsystem technologies. More specifically, materials property enhancements that are achievable as a result of having nanostructured MEMS components will be discussed along with the importance of property uniformity / microstructural integrity across the components. It should be noted that the results presented in this paper are from studies on electroplated nickel. Directionally similar results are expected for other metals used in MEMS, as already demonstrated for Co deposits ³.

Microstructural and Property Variations in Conventional Electrodeposits

Technical challenges with LIGA processes are numerous, and many of the performance / reliability problems can be attributed to the incommensurate scaling of microstructural features (i.e., grain size, grain shape, grain orientation) with the external dimensions of the components. It has been shown that many of the reliability and durability issues in electroplated microcomponents can be traced back to the microstructural evolution stages during the deposition process, leading to local property variations that point to a non-uniform structure within the material.

Figure 2 shows a cross-section of a nickel microcomponent prepared by conventional nickel electroplating technology. The grain structure changes from equiaxed fine grains to columnar having a high aspect ratio with the long axis in the growth direction. In other words, strong grain shape and size anisotropies exist within the deposit. This type of microstructure is one of the main drawbacks of LIGA products made by using conventional electroplating techniques, in particular when dealing with component and device sizes that are comparable to the size of the anisotropic (non-uniform) grains in the final product. Materials properties that are sensitive to grain orientation (e.g., elastic constants, thermal expansion, magnetostriction) would change from quasi-isotropic in the initial fine-grained deposit layer to being strongly anisotropic with increasing deposit thickness. Similarly, properties that depend strongly on grain size (e.g., hardness,

strength, ductility, coercivity) will show considerable changes in a direction perpendicular to the substrate.

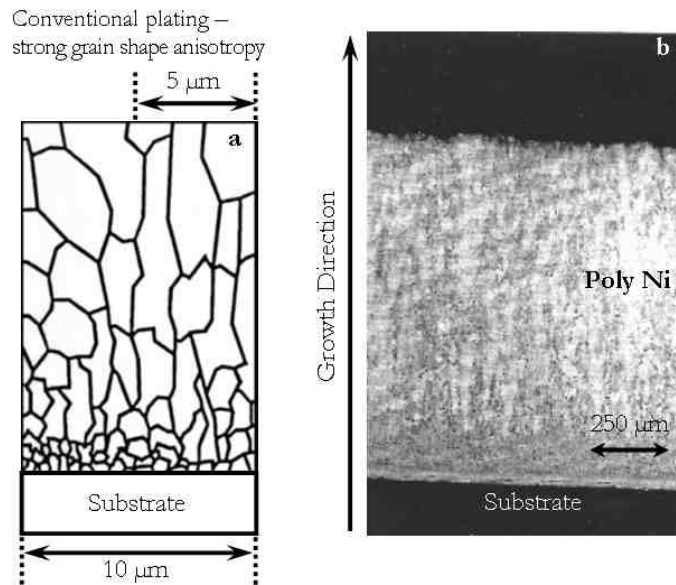
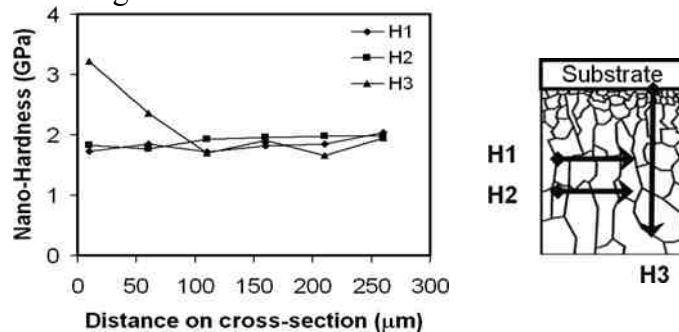


Figure 2 A microcomponent made of electroplated polycrystalline nickel produced by conventional electrodeposition: a) schematic cross-section of the columnar grain structure; b) actual cross-section [adopted from ref.4].

Figure 3 shows the results of nanoindentation measurements (nano-hardness and Young's modulus) performed on the cross-section of an electroplated nickel microcomponent produced by conventional means⁴. The results presented in this figure clearly show the inadequacy of such a microstructure. The properties depend on the location of the measurements and can show quite large variations. The hardness ranged from over 3GPa in the fine-grained region to less than 2GPa in the columnar grain section. The Young's modulus varied by a factor of more than 2 between the lowest and highest values (275GPa vs. 130GPa) depending on the local crystal orientation³. These observed non-uniformities present a serious issue in terms of device performance that must be addressed for the next generation of MEMS technology dealing with components in the micrometer range.



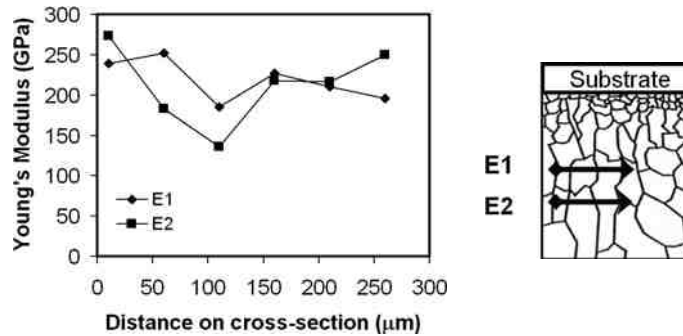


Figure 3 Cross-sectional hardness and Young's modulus for a nickel microcomponent produced by conventional electrodeposition showing non-uniformity across the component [adopted from ref.4].

Although efforts have been made to address and alleviate these concerns, primarily recognizing the importance of crystallographic texture and developing remedies such as post-deposition recrystallization annealing and crystallographic texture modifications during plating⁵, success using these approaches has been rather limited.

Microstructure and Properties of Nanostructured Electrodeposits

The importance of average grain size and grain size distribution in the final LIGA products has not yet been fully recognized and addressed. This is the main focus of our research efforts in terms of advancing MEMS technology. In this approach, the problem of non-uniform grain size and shape is alleviated by reducing the grain size of the electrodeposit to the nanometer range – by means of a nano-electroplating process^{6,7}. By this approach, fully dense nanostructured deposits can be produced with only minor modifications to existing electroplating processes. Figure 4 illustrates this principle. Figure 4a is a schematic diagram representing the nanometer-sized grains grown on a substrate with a very uniform grain size and shape distribution; Figure 4b is a cross-sectional transmission electron micrograph of an actual nano-electroplated nickel component. The nanostructure in the electrodeposit is established at the interface with the substrate and maintained throughout the entire thickness of the component.

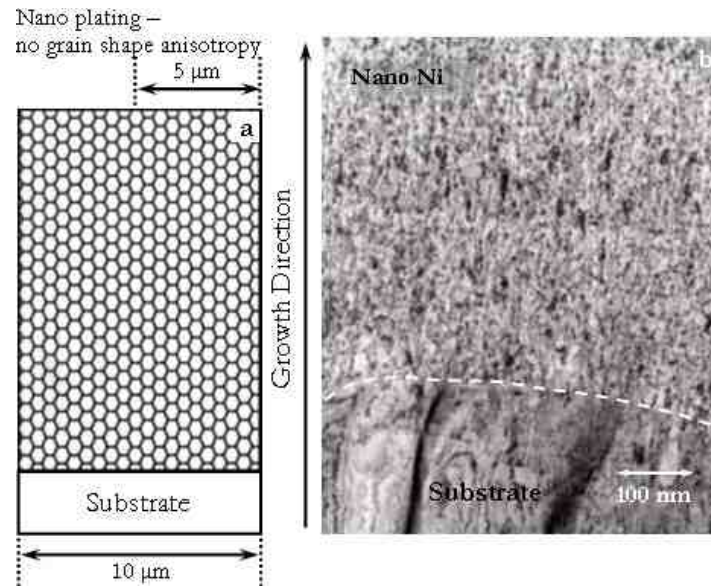


Figure 4 A microcomponent made of nanocrystalline nickel deposited onto polycrystalline phosphor bronze substrate by nano-electroplating: a) schematic cross-section showing uniform grain size and shape; b) actual cross-section [adopted from ref.4].

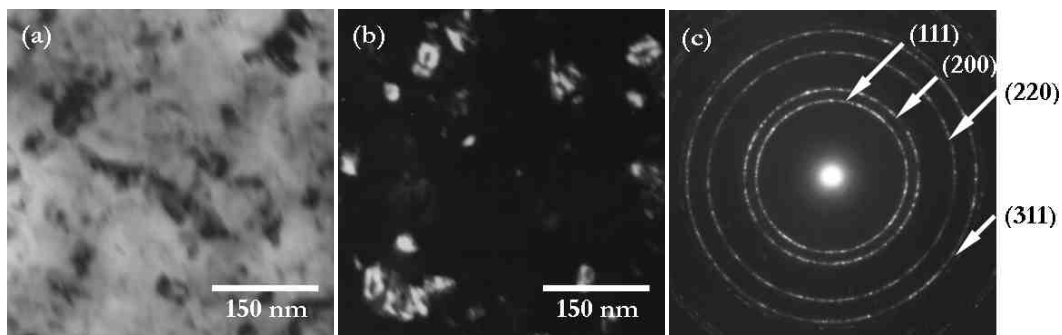


Figure 5 Electron micrographs: (a) brightfield; (b) darkfield) and electron diffraction pattern of an electroplated nanocrystalline pure nickel (25nm grain size).

Figure 5 shows the brightfield (BF) and darkfield (DF) transmission electron micrographs as well as an electron diffraction pattern (DP) of the electroplated nanocrystalline pure nickel sample in planar cross-section parallel to the substrate. It can be seen from the BF and DF micrographs (Figures 5a and 5b) that the plated material exhibits an equiaxed microstructure also in planar cross-section. The continuous rings seen in the electron DP (Figure 5c) show clearly that this deposit has a true nanocrystalline structure, with individual grains being separated by large angle grain boundaries.

The same cross-sectional mechanical properties (i.e., nano-hardness and Young's modulus) of nano-electroplated microcomponent materials have also been assessed (see Figure 6). When

examining these properties, two major improvements can be observed for nano-electroplated nickel. First, the local variations of hardness and Young's modulus seen in Figure 3 for conventional nickel are notably absent. There is no gradient in hardness in a direction perpendicular to the substrate, and the Young's modulus is relatively constant at a level of about 200GPa, the value for bulk nickel report in the literature⁸. Second, the overall hardness of the nano-plated component is considerably higher (> 6GPa) compared with the hardness of the conventional nickel (2 to 3GPa, depending on location in Figure 3). It is very encouraging to confirm that the uniform structure of nanocrystalline deposits indeed results in uniform and enhanced mechanical properties throughout the entire cross-section of the component. For the end user, nano-plating of MEMS components therefore minimizes the uncertainty regarding property variations in the final product.

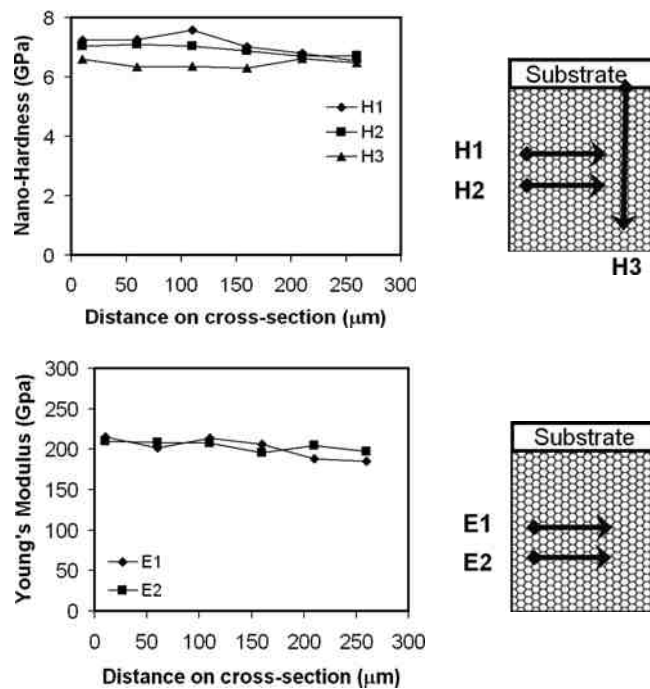


Figure 6 Cross-sectional nano-hardness and Young's modulus for a nickel microcomponent produced by nano-electroplating. Properties are relatively constant throughout the entire component [adopted from ref.4].

Effect of Grain Size on Properties of Nickel Electrodeposits

With the major issue of microstructural and property uniformity of nano-plated nickel addressed in the previous section, it is of interest to examine some of the other properties of nanocrystalline nickel that could be beneficial to applications in MEMS technology.

Properties Affected by Grain Size

Many properties have been shown to be strongly affected by the crystallite size of the material. For example, yield strength (σ_y), tensile strength (σ_{UTS}), hardness (H_V), tensile elongation (ϵ_f), wear rate (W) and coefficient of friction (μ), coercivity (H_C) and corrosion potential (E_{CORR}) have all been shown to display very different property values for nanocrystalline materials as compared to conventional polycrystals⁹. When considering materials properties important to MEMS applications, these grain size effects can offer additional advantages. The following discussion will focus on some of the more important property enhancements achieved in electroplated nanostructures.

Figure 7 shows the microhardness (H_V) of electroplated nickel of various grain sizes (from $10\mu\text{m}$ down to 10nm). At a grain size of $10\mu\text{m}$, the microhardness of conventional nickel is less than 200VHN . However, as grain size decreases, the hardness of the material increases as per the Hall-Patch relationship^{10,11}, reaching a hardness value that is over 300% that of the conventional material at a grain size of about 10nm .

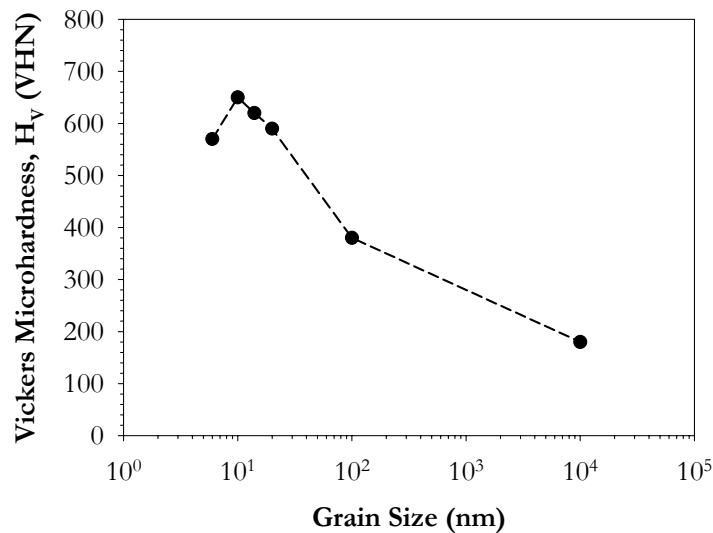


Figure 7 Vickers microhardness of electroplated nickel as a function of grain size showing a three-fold increase when grain size is reduced from $10\mu\text{m}$ to about 10nm ⁹.

It should be pointed out that there is a deviation from the regular Hall-Patch relationship at the smallest grain sizes ($< 10\text{nm}$) for nano-electroplated nickel. The reasons for this deviation which is also observed in some nanocrystalline materials produced by other synthesis methods are still not completely understood. While various theories and models have been proposed, a detailed discussion of this effect is beyond the scope of this paper.

Figure 8 shows a similar grain-size dependence for the yield strength of nickel. The yield strength of conventional nickel, at a grain size of $10\mu\text{m}$, is about 180MPa . It increases with decreasing grain size reaching a maximum value of $> 900\text{MPa}$ at 10nm .

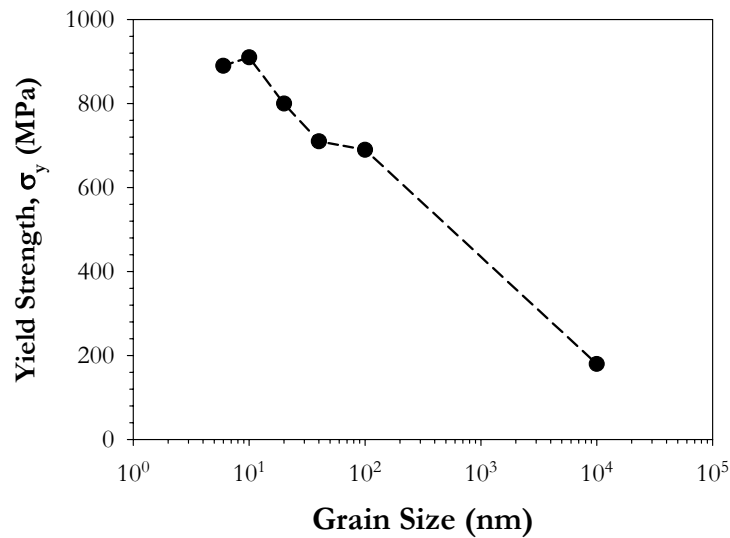


Figure 8 Yield strength of electroplated nickel as a function of grain size showing a five-fold increase when grain size is reduced from $10\mu\text{m}$ to about 10nm ⁹.

While hardness and yield strength are static mechanical properties, wear can be considered a kinetic property of particular importance for MEMS structures involving moving parts. In Figure 9, the sliding wear rate of nickel is presented as a function of grain size. As grain size decreases, the wear rate dropped substantially. The wear rate for a 10nm nano-electroplated nickel is less than 1% that for the conventional material at $10\mu\text{m}$ grain size.

Since grain boundaries and triple junctions contribute to the scattering of electrons, a nanostructured material is expected to have a much higher electrical resistivity simply because of the high population of these intercrystalline defects in the structure. For example, at a grain size of 10nm , and assuming a grain boundary width of 1nm as proposed in a recent geometrical model¹², the fraction of atoms that are located either at the grain boundaries or triple junction is over 30%. In other words, about one-third of the entire material consists of interfacial defects.

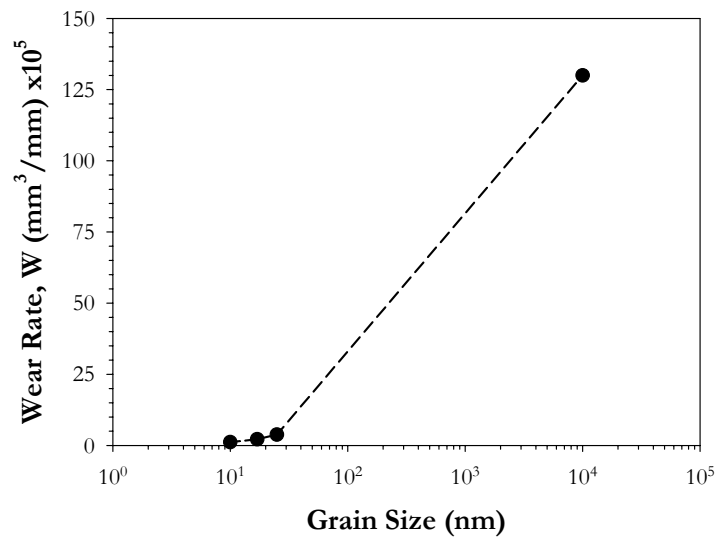


Figure 9 Sliding wear rate of electroplated nickel as a function of grain size; 10nm nano-electroplated nickel showing less than 1% of the wear rate of 10 μ m conventional nickel⁹.

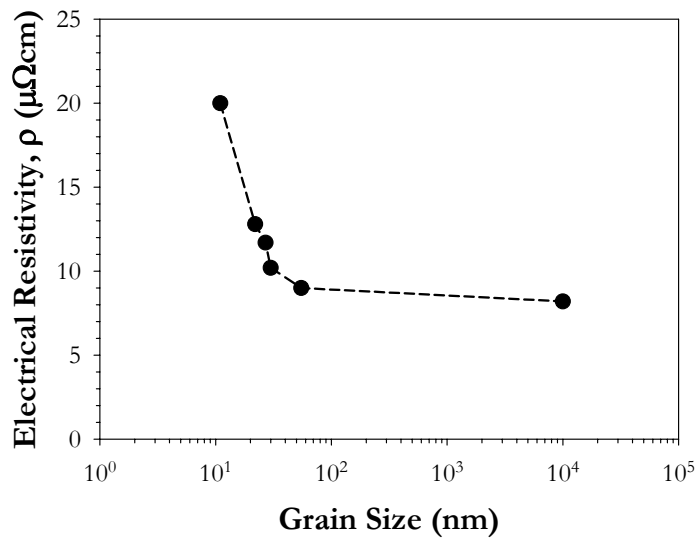


Figure 10 Room temperature electrical resistivity of nickel as a function of grain size, showing strong grain size dependence below 100nm⁹.

Electrical resistivity measurements were performed on electrodeposited nickel with various grain sizes. As seen in Figure 10 for nano-electroplated nickel, the room temperature electrical resistivity is increased by about 250% for a material having a grain size of 10nm ($\rho \approx 20\mu\Omega\text{cm}$) when compared with conventional nickel with a grain size of about 10 μ m ($\rho \approx 8\mu\Omega\text{cm}$).

Grain Size Independent Properties

There are several properties of electroplated nickel that are not or very little affected by grain size including heat capacity (C_p), saturation magnetization (M_s), thermal expansion coefficient (α) and Young's modulus (E)⁹. The following discussion will be limited to the most important properties considered in the material selection for MEMS applications.

The published value of Young's modulus (E) for pure polycrystalline nickel is about 207GPa⁸. In Figure 11, Young's modulus measurements for electrodeposited nickel are plotted as a function of grain size from 10 μ m to less than 10nm. The value for the large-grained sample agrees very well with the published value. The graph shows that Young's modulus is essentially independent of grain size.

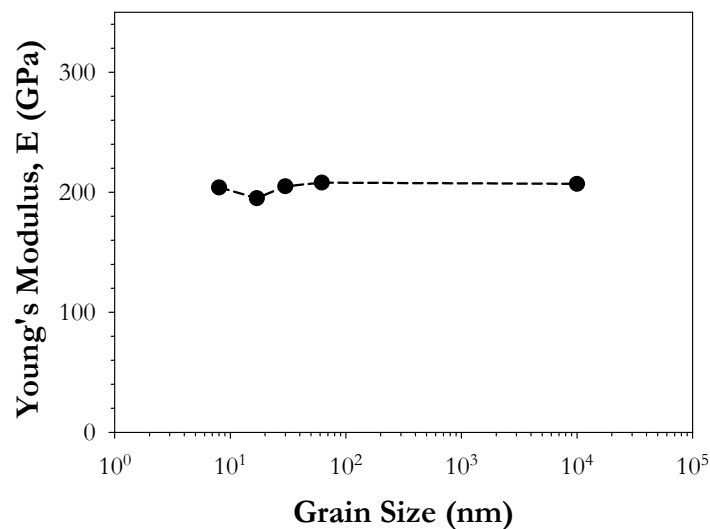


Figure 11 Young's modulus of nickel as a function of grain size⁹.

Another property that is not strongly affected by an ultra-fine grain structure is the thermal expansion coefficient, α . Figure 12 shows that, compared with the thermal expansion coefficient of nickel at 10 μ m grain size, nickel with a 10nm grain size shows only a few percent ($\approx 3.5\%$) reduction.

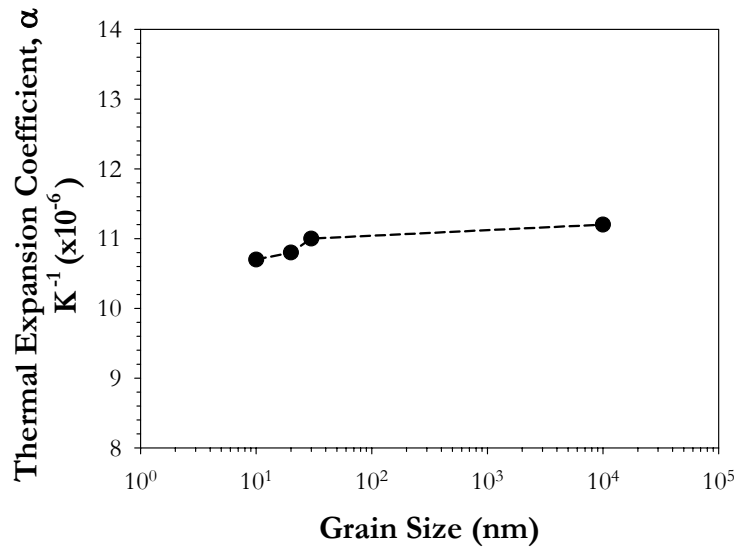


Figure 12 Weak grain size dependence for the thermal expansion coefficient of nickel⁹.

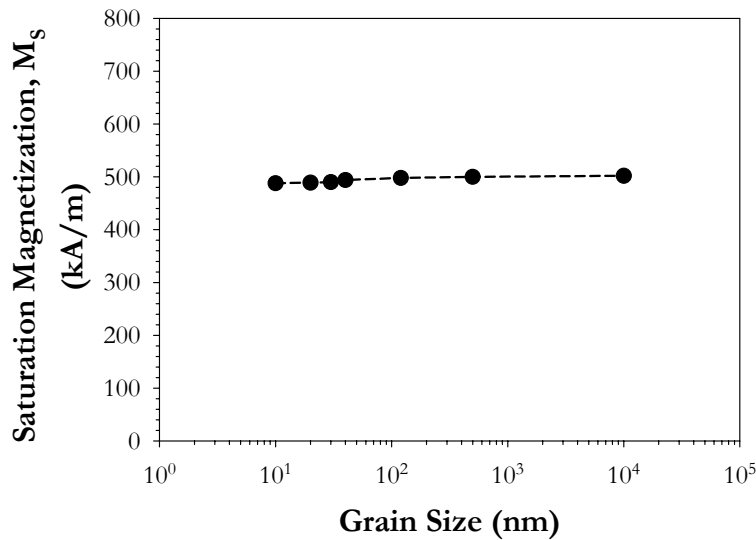


Figure 13 Saturation magnetization of electrodeposited nickel showing no significant grain size dependence⁹.

One of the earliest studies on the saturation magnetization (M_s) of nanocrystalline nickel (< 20nm grain size) prepared by the inert gas condensation technique found a 40% reduction in M_s compared to conventional nickel¹³. This unfavourable result initially caused some concerns in terms of full functionality of nanocrystalline materials in magnetic applications. However, this drastic decrease in saturation magnetization was later attributed to residual porosity resulting

from this particular processing method. When fully-dense nano-electroplated nickel was examined, it was found that the saturation magnetization is not strongly affected by grain size¹⁴. Figure 13 shows this result; the saturation magnetization for pure nickel remained the same throughout the entire grain size range examined (from 10 μ m to about 10nm). For MEMS components requiring soft magnetic materials, this result is of significant importance.

Performance Enhancement by Grain Size Reduction

In addition to offering a fundamental solution to eliminate unacceptable property variations in MEMS components, electroplated nanostructures exhibit inherent property enhancements that are beneficial in the final application of MEMS devices. Given the information available on property enhancements for nano-electroplated nickel presented above, one can assess performance improvements that can be expected from nanostructured MEMS components. In this section, some important performance indicators for microcomponents will be explored using an Ashby-type approach¹⁵.

With the ever-shrinking dimensions of MEMS structures, the strength of miniature components becomes increasingly important. As seen in Figure 8, the yield strength of nanostructured nickel (at a grain size of 10nm) was increased by a factor of 5 over that of a large-grained nickel material (10 μ m). This structurally superior material is able to withstand much more stringent loading conditions than what can be tolerated with conventional materials. From a deformation mechanism point of view, the advantage of nanostructured materials is enormous when considering components with very small external dimensions as indicated in Figures 3 and 4, for example, for a hypothetical component with a width of 5 μ m. For such a small component, the width approaches the grain size of conventional nickel (see Figure 3). Therefore, there are very few grain boundaries in the cross-section of such a component which could prevent dislocation motion, resulting in a rapid loss of strength. On the other hand, for a nanocrystalline component with the same external size (see Figure 4), there are still hundreds of grain boundaries in cross-section to maintain the strength level of the system.

Other materials properties that are dependent on the yield strength will also benefit from this substantial increase in the yield strength. For example, resilience, R (or elastic energy storage capacity), is one of the properties that depend on both yield strength and Young's modulus: $R = \sigma_y^2/2E$. It is one of the most important properties in components such as micro-springs and levers. With the improved yield strength, nano-nickel can store much more elastic energy than conventional materials. Figure 14 shows the resilience of nickel as a function of grain size, normalized with respect to the value obtained for a 10 μ m nickel material. Simply by reducing the grain size to 10nm and keeping all other factors constant, the resilience of nickel is enhanced by a factor of 25 over that of conventional material.

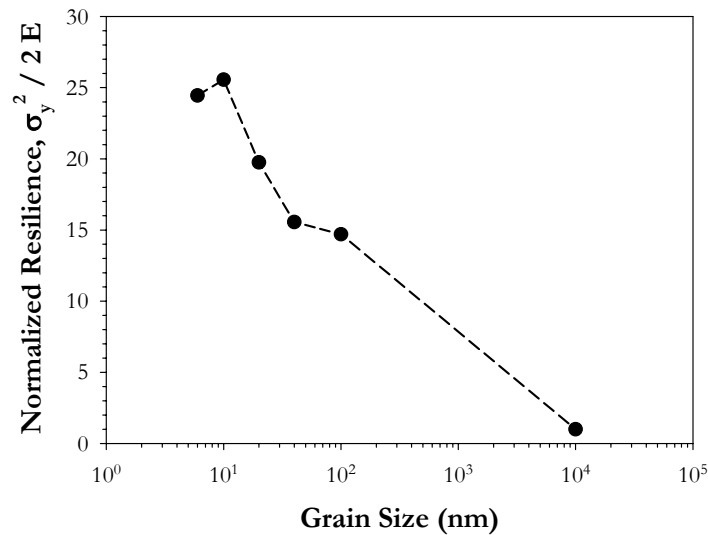


Figure 14 Resilience of nickel as a function of grain size (normalized to value at 10 μ m grain size).

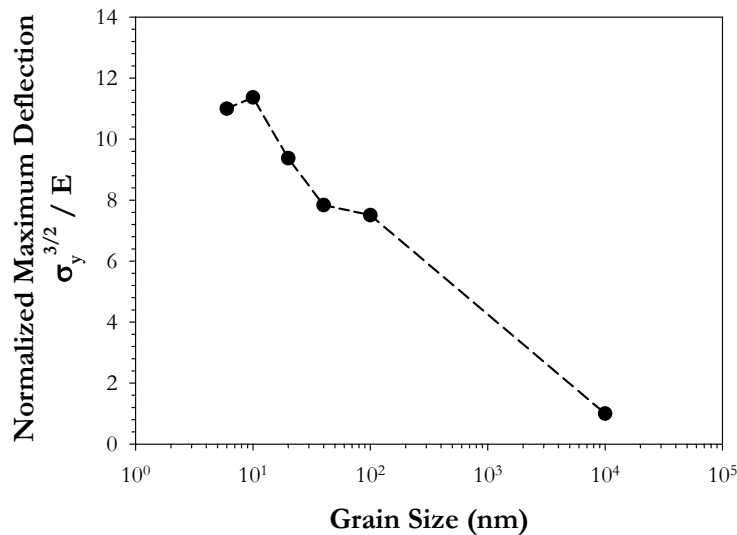


Figure 15 Flexural Strength of nickel as a function of grain size (normalized to value at 10 μ m grain size).

Similarly, for diaphragm or membrane type applications, the maximum deflection under a specific pressure before fracture occurs also depends on yield strength and Young's modulus. In this case, the performance indicator of importance is $M = \sigma_y^{3/2}/E$, with maximum deflection being proportional to M . Figure 15 shows M of nickel as a function of grain size. As seen in this figure, the maximum deflection attainable for a nano-electroplated nickel is almost 12 times higher than for conventional nickel material.

From the performance enhancement seen in hardness, resilience and maximum deflection for a specific pressure, microcomponents such as micro-springs, levers or diaphragms can operate at a higher load levels and more demanding conditions without failure. Conversely, when designing a MEMS device using nano-electroplated nickel, the same specifications can be met with a smaller component, which would create more real estate for other components / devices on a platform where space is a premium.

Another enhancement that has been observed for nano-electroplated nickel is the sliding wear characteristics, with sliding wear resistance being the reciprocal of sliding wear ($1/W$). The sliding wear resistance of nickel is plotted as a function of grain size, normalized to the value obtained for the $10\mu\text{m}$ material (see Figure 16). For nano-electroplated nickel at 10nm grain size, the sliding wear resistance increases by more than 100 times. With this magnitude of improvement, the life of the microcomponents will be considerably prolonged. In other words, dimensional degradation due to wear of the already small microcomponents will be greatly reduced.

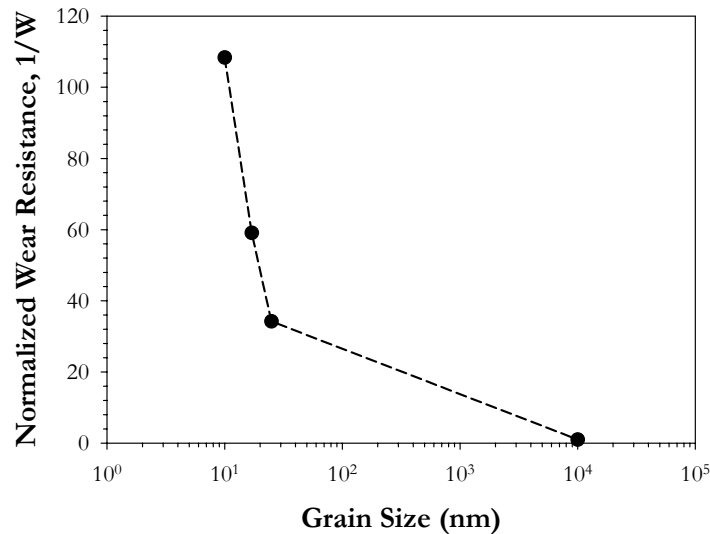


Figure 16 Sliding wear resistance of nickel as a function of grain size (normalized to the value at $10\mu\text{m}$ grain size).

Energy losses due to eddy currents can lead to diminished efficiency of an electromagnetic component / device. A material having a higher electrical resistivity (ρ) reduces eddy current losses (which are proportional to $1/\rho^2$) and improves overall energy conversion efficiency. This property is particularly important in high frequency applications. In conventional materials, higher resistivities are usually achieved by incorporating solute elements into the material by design. However, magnetic properties that are sensitive to solute additions (e.g., saturation magnetization, magnetostriction and coercivity) are often compromised in this approach. In light of this, the ideal solution would be to have a material with an inherently higher electrical resistivity without the need for changing its composition. From the enhanced electrical resistivity seen for

nano-electroplated nickel (see Figure 10), nominal energy losses can be estimated. Figure 17 is the energy loss through eddy currents as a function of grain size for nickel, normalized with respect to conventional nickel with 10 μ m grain size. The nano-electroplated nickel exhibits a loss that is less than 20% of that of the conventional material. This provides for more efficient devices and the amount of work that can be expected from a device will be improved compared with those made of conventional materials. Furthermore, as the energy loss translates to heat, the size of heat sinks can be reduced and requirements in thermal conditioning and device environmental control can be relaxed considerably.

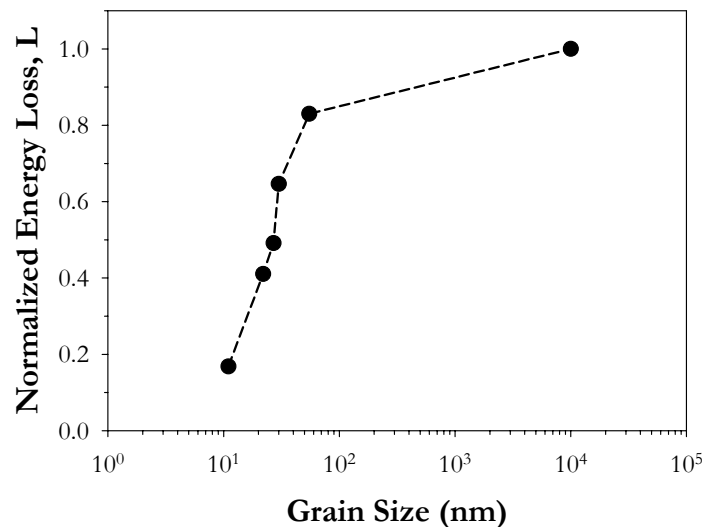


Figure 17 Energy loss due to eddy currents as a function of grain size (normalized to value at 10 μ m grain size).

Summary

Although MEMS technology has greatly advanced in recent years, there are still several technical problems associated with LIGA-processed materials. The non-uniformities in microstructure observed across conventional LIGA microcomponents result in considerable local property variations that must be addressed, as they become intolerable with the ever-decreasing size and dimensions of the microcomponents.

We have presented a new approach to electroplating-based MEMS technology by which proper microstructure scaling is taken into consideration to ensure materials property stability and microstructural integrity throughout the microcomponents. By nano-electroplating, no point-to-point property variations in cross-sections of nickel microcomponents were eliminated. Through nano-electroplating, many materials properties are significantly improved, including hardness, yield strength and wear resistance. Moreover, when compared to conventionally prepared materials, nano-electroplated nickel is over 25 times more resilient, 12 times higher in maximum deflection, over 100 times more wear resistant and exhibits 80% less energy loss due

to eddy currents. From this nanostructured materials platform, the next generation of performance enhanced MEMS components and devices can be developed.

Acknowledgements

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