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Effect of Ppm Level Dopant on Ductility of Ultrafine Grained Gold Wires

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Abstract. The addition of calcium (Ca) simultaneously improves the ductility and strength of UFG Au wires. Based on the observation on stacking faults, microstructures, simulation results and significant effect of Ca on grain boundary related properties, it is inferred that segregation of Ca to stacking faults and grain boundaries has occurred to induce effective stacking fault energy (SFE) reduction and properties improvement. Considering the known greater impact of SFE in UFG/ NC metals, segregating dopants are proposed to be an effective strategy for achieving dual improvement in this class of materials. Also, dopant selection criteria for this purpose is also suggested and verified.

Introduction

In recent years, bulk nanocrystalline (NC) and ultrafine-grained (UFG) metals and alloys have attracted a great deal of attention as potential candidates for next-generation structural materials. Indeed, success has been achieved on the strength improvement with grain size refinement to nano-scale, extending from the well-known Hall-Petch relation (at least down to ~ 20 nm) [1,2]. However, in general it is reported that the ductility of NC/UFG metals is much lower than that of their CG counterparts, even for metals that are extremely ductile in CG form. For example, CG copper (Cu) can have elongation (EL) as large as ~ 60%, while EL of Cu (100 nm > grain size > 10 nm) reported ranged from 1% to 7% [3, 4]. Low ductility poses an insurmountable hurdle in manufacturing process because of formability issues. Therefore, strategies to attain both high strength and ductility (dual improvement) in NC/ UFG materials need to be developed, a goal shared by manufacturers of UFG gold (Au) bonding wires in microelectronic industry. Most of the current efforts done are through manipulating the process. In contrast, work on the effects of alloying/ doping in UFG/ NC materials are limited [5]. And in most cases, the effects of alloying at higher concentration (not doping) which in general lead to second phase formation are studied [6, 7, 8]. This paper focuses on a much lower dopant concentration in the ppm level, for which information is clearly lacking.

Zhao et al. has demonstrated the success of achieving dual improvement, by reducing the stacking fault energy (SFE) of copper via the addition of 10% zinc [9]. However, this approach can not be implemented on Au bonding wires, since the doping levels are required to be low for consideration on corrosion resistance, electrical conductivity etc. It is known that alloying (in solid solution form) typically decreases SFE of the matrix materials. The usual range of alloying study is from few percent up to as high as ~ 40 (weight) wt. % [10], but there are also studies which focused on lower alloying concentration of as low as 0.2 wt. % [11]. The mechanism behind the phenomenon is the segregation of dopant to stacking fault (SF), stabilizing SF and lowering SFE, as first pointed out by Suzuki. The absorption phenomenon can be treated in the same manner as at grain boundaries (GB), and are expressed in terms of the Gibbs adsorption isotherm here [12]:

$$\left(-\frac{\partial SFE}{\partial G_2}\right)_T = \Gamma_2 - \frac{x_2}{x_1}\Gamma_1 \tag{1}$$

where x_2 and x_1 are the atom fraction of solute and solvent in the bulk phase, G_2 is the chemical potential of solute and Γ_2 and Γ_1 are the surface excess concentration of solute and solvent at the fault. For dilute solutions, where $\Gamma_2 > (x_2/x_1)\,\Gamma_1$, the adsorption isotherm reduces to:

$$\left(-\frac{\partial SFE}{\partial G_2}\right)_T = \Gamma_2 \tag{2}$$

Eq. 2 shows that when solutes accumulate at the SF, the equilibrium SFE decreases. It is reasonable to assume that dopants with strong segregation tendency can be much more effective in reducing the SFE of materials, compared to dopants that are well accommodated in the matrix. The objective of this paper is to explore the effect of segregating dopant on the mechanical and microstructural properties of UFG Au bonding wires. Calcium (Ca) is chosen as it is a well-known strengthener in gold bonding wire industry [13]. This element is also selected for its potential to effectively reduce SFE of Au wires, as it has low solid solubility in Au [14] and has high valency [15].

Experimental

The experimental procedure used herein is described as follows. First, for the preparation of the wire sample, pure Au (99.999%) was doped with Ca at the levels of 20, 60, 90, 250 and 500 ppm by wt. The doped Au ingots were manufactured using a continuous casting process under an inert gas atmosphere. Each ingot was drawn into 25 μm diameter wires using a conventional multi-stage wire drawing process under identical process conditions at room temperature. The reduction ratio from ingot to final diameter was ~ 99.99%. In the discussion that follows, we have labeled them as Au250Ca for Au wires with 250 ppm Ca, for example. For tensile test, an INSTRON 5543 universal tensile testing machine was used, with initial gauge length of ~25 cm and a crosshead speed of 10^{-2} s^{-1}, following ASTM standard [16]. To observe the microstructure, transmission electron microscopy (TEM) was employed. Sample preparation was done with FEI FIB200XP focused ion beam (FIB), to thin down the wire. Standard copper grid was cut into a half and used as a holder for wire. A JEM-2100F TEM was used to study the microstructure. Multiple areas from at least three samples were analyzed for each composition.

Results and Discussions

Tensile Properties. Room temperature tensile properties of as-drawn Au wires at several levels of Ca are compared in this section. Fig. 1 shows the engineering stress strain curves of Au wires at five levels of Ca additions, from 20 ppm to 500 ppm. The stress strain curve of Au5N wire is included as a reference. Even in the as-drawn state, Au5N has good ductility of ~3.5%, but has extremely low strength of ~ 100 MPa, which is one of the reasons why doping is needed for Au bonding wires. By adding only 20 ppm of Ca, the strength goes up to ~400 MPa, but the ductility is also significantly reduced to ~1.6%. The strength of Au wire is as high as ~700 MPa, at the highest Ca level of 500 ppm used. Ca is a well-known strengthener used in Au bonding wire industry, with their strengthening effects documented in many patents [13]. Interestingly, Ca also has an obvious beneficial effect on the ductility of Au wires. It has been reported in one patent that Ca can prevent brittleness [17]. From the results, it is clear that both ductility and strength of Au wires are progressively and consistently improved by Ca. However, ductility improvement seems to saturate and stop at Au500Ca. For this reason, subsequent study will focus only on wires with 250 ppm Ca and below.

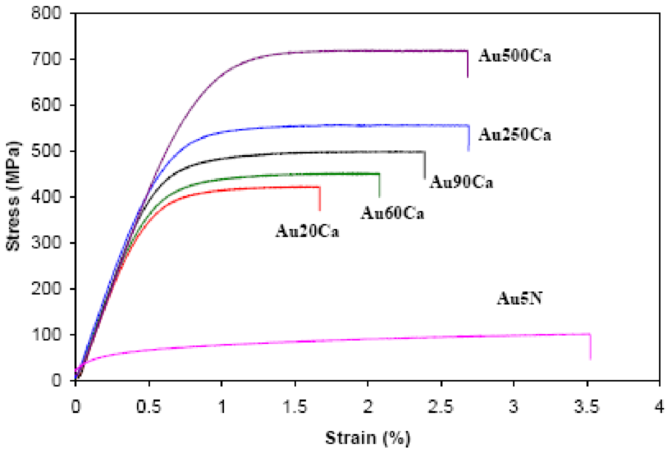

Fig. 1. Engineering tensile stress strain curves of as-drawn Au wires at various levels of Ca.

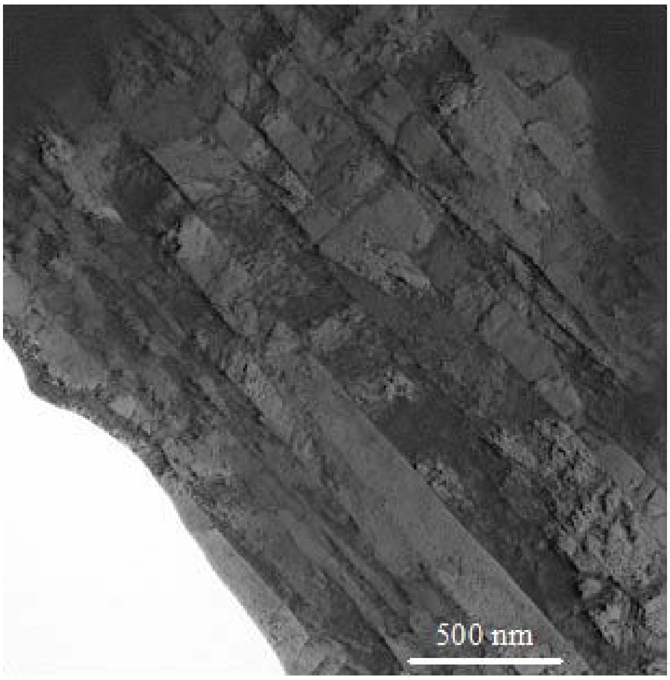

Fig. 2. Typical microstructure of Au wire, shown here is Au250Ca.

Microstructures. No second phase is observed in Au wires, with doping concentration of 20 ppm to 250 ppm. However, second phase starts to appear at 500 ppm Ca gold wire [18]. This suggests that Ca stays as solid solution below 250 ppm Ca. The phase diagram of Au-Ca shows a maximum solid solubility of 3750 ppm by wt. at ~800°C, but the solid solubility of Ca in Au at room temperature is not known exactly [19]. Based on the preferential segregation tendency of Ca, it is predicted that most Ca atoms could have segregated to the SF and GB in Au. Fig. 2 shows the bright field TEM image of typical microstructures of Au wires. The grains have elongated, fiber-like structures along the drawing direction, which is a characteristic of wires that have under went extensive drawing process. The fiber grains have diameter between 100- 500 nm, which Au wire can be considered as UFG and NC metals.

The theory of dopant segregation to SF to cause SFE reduction in CG metals is fairly complete, however direct chemical evidence for this type of segregation is generally lacking, because of the abundance of SF and difficulty of microanalysis [15]. While only several investigators were able to directly identify the segregation [20, 21], most studies presented indirect identification. For example, changes in the width of an extended dislocation in a Cu–8 at. % silicon alloy was inferred to be due to Suzuki segregation [22]. In our case, even though we did not directly identify Ca segregation to SF/ GB, there are several strong indications of that:

1. Mostly the segregation effect manifests itself as an increment in the stacking fault width (SFW), which is one out of the two established methods for SFE estimation [12, 23]. We have shown that as in the case of Ref. 22, indeed, SFW and SF density in Au wires were increased by Ca addition [24], which signifies SFE reduction.
2. Along with the direct evidence from point 1, the observations of a straighter slip mode, more evenly distributed dislocation network, higher dislocation density and increasing random texture are also in agreement with the expected microstructures with lower SFE [18]. This also confirms that Ca has induced SFE reduction in Au wires at these low ppm levels [24]. The fact that SFE reduction can be accomplished by ppm level of doping suggests that solid solution alone of Ca in Au wire is not likely to be the scenario.
3. Ab initio simulation has predicted high segregation tendency of Ca in Au. The segregation energy obtained (117 kJ/ mol) [24] is comparable to the segregation energy of known segregating systems (~100 kJ/mol) [25].
4. Ca was observed to have strong impact on creep [18] and Hall-Petch coefficient [26]. These obvious impacts on GB-related properties have suggested the segregation tendency of Ca in Au wires.

The SFE reduction as induced by Ca doping is suggested to bring about simultaneous strength and ductility improvement, as process remains identical for the wires. Large amount of very fine nano-twins, which can be as thin as ~ 1 Å (equivalent to two layers of SF) are also observed in Au250Ca (Fig. 3), while these features are not seen on Au wires with lower Ca concentration as a result of higher SFE. Fig. 3c is a typical SAD pattern taken using <110> zone axis from the area containing twins, which shows the mirror spots appear wrt the {111} plane. Also, these twins are observed to nucleate from the GB or GB junctions in Au250Ca. In Fig. 3a, the red lines delineate the boundaries of grains. This suggests that twin nucleation occurs via a mechanism unique to UFG/ NC metals, which is partial dislocation nucleation from the GB. This differs from the usual pole mechanism for twin nucleation in CG metals, whereby twinning is usually form in the area with highly localized stress, such as the intersection of shear bands [27, 28]. Even though the twin density can be abundant in some areas, their appearance is localized, i.e. they are not ubiquitous but concentrated in certain areas, likely those containing high local stress.

It is postulated that dopants that segregate to GB can also reduce the SFE in the case of UFG/ NC metals, on top of the segregation to SF as in CG materials. It is known that in CG metals, dopants that segregate to GB can create ledges which are conducive to dislocation generation [15]. Similar scenarios can happen in UFG or NC materials, but probably partial dislocations instead of full dislocations shall be emitted, as dictated by the GS. It follows that segregation of Ca to the GB in UFG Au wires can encourage the emission of partial dislocations (giving SF) from the GB, where the ease of SF formation is a sign that SFE has been reduced [29]. Fig. 4 shows the HRTEM of Au250Ca. It can be seen that there is a ledge at the GB, and a twin is generated from the ledge, which could correspond to what was described earlier. Also, the localization of twin formation in Au wires supports the deduction that the areas with SF/ twin could be the area where the Ca is concentrated at. Ca first segregates to the GB to accelerate the partial dislocation nucleation process, and also segregates to SF to stabilize their existence. Therefore, through this double enhancement route, segregating dopant could have a more obvious impact on SFE. Also, it was reported that the impact of SFE on material properties of UFG/ NC materials will be larger than in CG metals [29]. The hardness of 2 mm Au rod with larger GS experienced ~20% hardness improvement with 90 ppm of added Ca [30]. For Au wires with very fine grain size, tensile strength is increased by 400% with the same level of Ca addition. The results imply that segregating dopants may have a greater role in determining the mechanical properties of UFG and NC metal than their CG counterpart.

Dopant Selection Criteria. We have demonstrated the significant impact of dopant segregation on SFE, and ultimately mechanical properties of UFG/ NC metals. In this section, a set of criteria is proposed for the selection of dopant with tendency to segregate and benefit both ductility and strength of UFG/ NC materials.

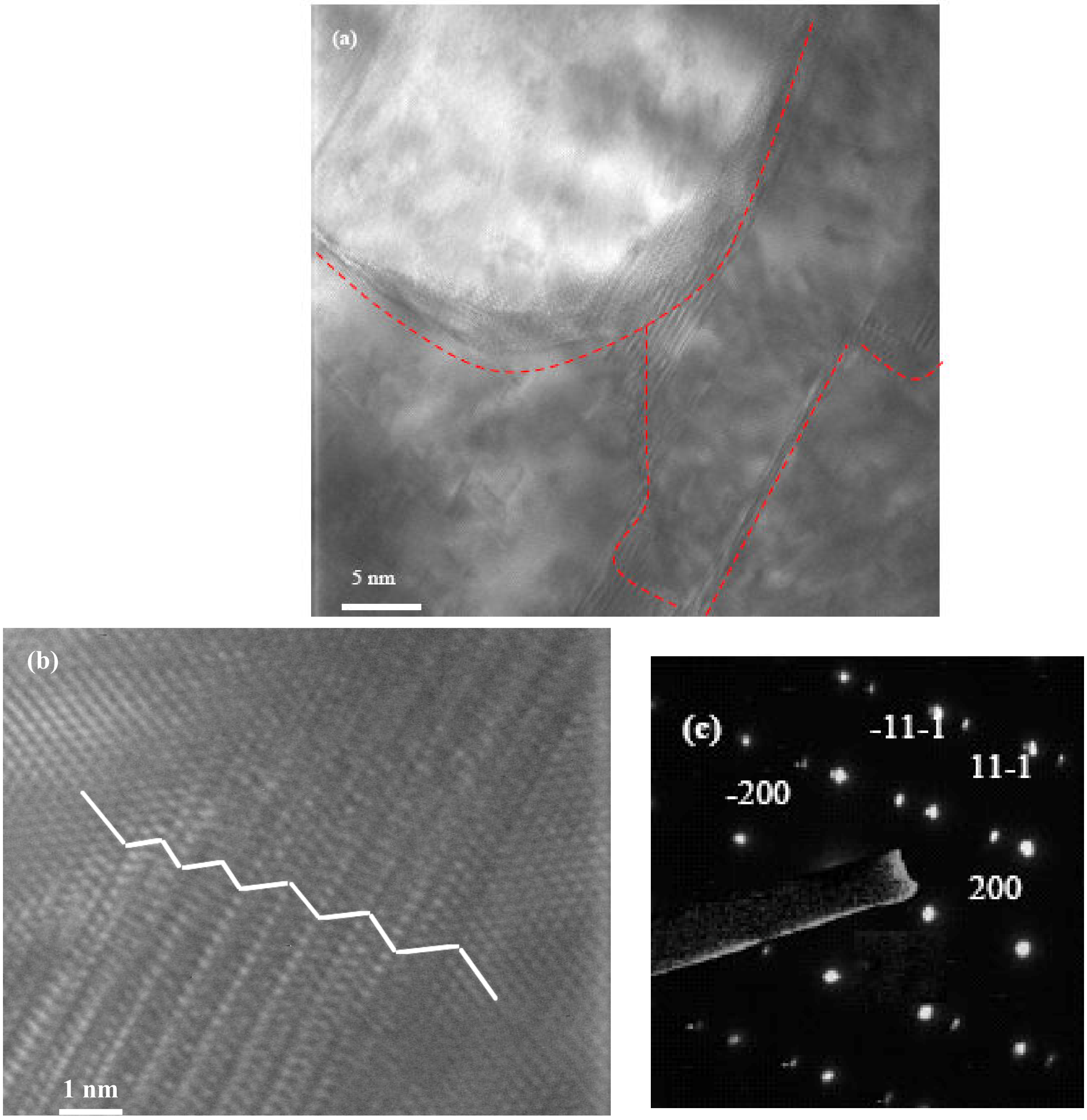

Fig. 3. TEM image of Au250Ca showing (a) multiple twins originating from grain boundaries; (b) HRTEM of the twins and (c) typical diffraction patterns obtained from twins.

Dopant Selection Criteria for Influencing Stacking Fault Energy: The initial hypothesis that Ca can reduce the SFE of Au wires effectively has been proven in this work. Therefore the establishment of criteria should start from the impact of dopant on the SFE of materials. It is known that SFE of a metal is generally lowered by alloying, while the effectiveness of the solute will depend on the solid solubility and valency of the solute. A simple expression has been derived relating SFE and alloying concentration:

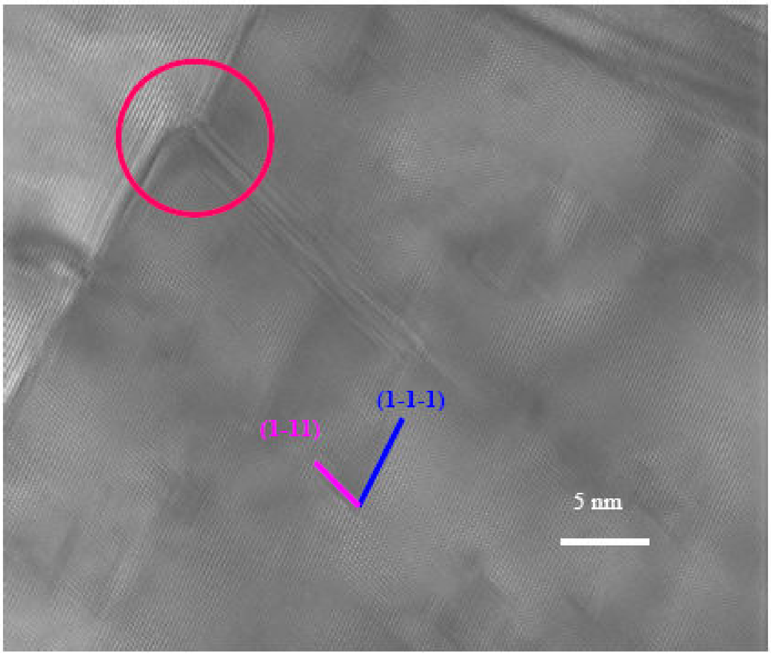

Fig. 4. TEM images of Au250Ca showing twin generation from a ledge on the GB.

$$In\frac{SFE_{alloy}}{SFE_{solvent}} = \delta[\frac{c}{(1+c)}]^2 \quad (5)$$

where $SFE_{solvent}$ represents the SFE of the pure metal (solvent) and SFE_{alloy} represents the SFE of the alloy, δ is a constant, and c = x/ x', with x is defined as the solute concentration and x' the solid solubility limit at high temperature [15]. From here, it can be seen that SFE of the base metal can be reduced more if the solid solubility of the solute is low in the metal. Solid solubility can be influenced by several factors, as suggested by Hume-Rothery's and Darken-Gurry's Rules [31, 32]: differences in atomic sizes, electronegativity and valency. Second, it is also known that SFE reduction is more effective with elements of higher valency, i.e. having higher numbers of electrons in the outermost energy level of an atom [14]. Therefore for the consideration of impact of dopant on SFE of metals, the parameters of concern are solid solubility, valency, size and electronegativity.

Dopants Criteria for Segregation Tendency: To have an indication of whether an element has the tendency to segregate, one need to look at the segregation energy (E_{seg}) of the dopant. E_{seg} is the difference in binding energy at T = 0 K of a dopant atom at a GB/ SF site versus one at a bulk site [33]. As segregation energy is not available in the literature, we have used simulation method to deduce this parameter [24].

Influence of Dopants on Grain Boundary Cohesion: Segregation of dopants can be beneficial or detrimental to the mechanical properties of the base materials, depending on their impact on GB cohesion (G_{coh}) [34]. Again, because this parameter is not available on the open literature, simulation was used as a mean to deduce the parameter [35]. The above mentioned criteria are listed in Table 1.

Besides Ca, platinum (Pt) and beryllium (Be) are also included for comparison (Pt and Be are also common dopants used in the wire bonding industry). Those properties that are highlighted in purple refer to the properties that have fulfilled the criteria. Out of the nine suggested criteria, Ca has satisfied seven of them. On the other hand, Pt which is known to have negligible impact on Au wire properties (at low concentration) does not fulfill any of the criteria [36]. Therefore it has been demonstrated that the proposed criteria work well in these two examples. It can be seen from the

SFE portion that the criteria converge to a requirement of dopant with low solid solubility. The applicability range of the suggested criteria has the restriction of dopant concentration, i.e. the concentration must be below their solid solubility, above which precipitation should occur.

Also, Be is noticed to fulfill all the criteria well. To examine the applicability of the model, Au wires were doped with Be to determine if the predicted concurrent strengthening and ductilization effect will be realized. Stress strain curves of the two wires with different levels of Be are shown in Fig. 5. As in the notation for Ca-doped Au wires, Au10Be and Au15Be refer to Au wires with 10 ppm Be and 15 ppm Be respectively. It is seen that with additional 5 ppm of Be, both ductility and strength of Au wires gets higher. This result is in agreement with a previous report, in which Brenner et al. have reported the beneficial effect of Be on both strength and ductility of Au wires in the 60's [37].

Table 1 Summary of properties with respect to the proposed criteria for dual improvement in Au wires

		Criteria	Ca	Be	Pt	Au
SFE	Solid solubility (at %)	The lower the better	1.8 @ 800 °C	0.2 @ 580 °C	complete solid solubility	N.A.
	Δ radius (%)	> 15%	35	-23	-6%	N.A.
	Δ electronegativity	> 0.4	-1.54	-0.97	-0.34	N.A.
	Crystal structure	Different	Same	Different	Same	N.A.
	Valence	The higher the better	2	2	1	1
GB segregation	Segregation energy	The higher the better	117	87	61	N.A.
GB cohesion	Strengthening energy	The more negative the better	-0.11	-1.61	1.25	N.A.
	Radius (Å)	The smaller the better	1.97	1.12	1.39	1.46
	Electronegativity	The lower the better	1	1.57	2.28	2.54

Conclusions

In this work, the effects of Ca on the mechanical and microstructural properties of UFG Au wires were investigated. Based on the observation, conclusions are summarized as below:

1. The addition of Ca in UFG Au wires simultaneously and progressively improves strength and ductility of Au wires, via effective SFE reduction at low ppm level.

2. Based on statistical measurement on SF width/ density as well as the observed microstructure, high segregation energy obtained and significant impact of Ca on creep rate and Hall-Petch coefficient, it is deduced that segregation of Ca to SF and GB has occurred in Au wires.
3. As there are localized, abundant partial dislocations and twins originating from GB, it is proposed that on top of dopant segregation to SF (as in the case of coarse-grained (CG) metals), segregation to GB in UFG and NC metals could also enhance the effectiveness of SFE reduction. This double effect will lead to efficient SFE reduction with segregating dopants in UFG/ NC metals. It is also known that the effect of SFE is more pronounced in UFG/ NC metals than in CG, the result is that Ca has a disproportionately larger effect on the mechanical properties of UFG Au wires at low concentration. It is seen that the effect of doping on strength is ~ 20 times more in UFG Au wires, compared to Au rod. The results imply that the effectiveness of segregating dopant in reducing SFE can be used as a strategy for the accomplishment of dual improvement in UFG/ NC materials.
4. The dopant selection criteria proposed here centers on the impact of dopant on SFE. Since segregation tendency is found to be relevant, segregation energy and the effect on GB cohesion upon segregation are examined. Requirements on six materials parameters are suggested: atomic size misfit, crystal structure, valency, electronegativity, segregation energy and strengthening energy, some of which are established via ab initio simulation within the framework of density functional theory.
5. The proposed criteria for dopant selection to achieve dual improvement have been experimentally verified in two additional systems: gold-beryllium and gold-platinum. Both calcium and beryllium have fulfilled the criteria and were demonstrated to bring about the unique effect of simultaneously increasing the strength and ductility of Au wires, in agreement with published patents. Platinum on the other hand, does not conform to the guidelines, and it shows insignificant impact on wire properties at the ppm level. These criteria are limited to dopant concentration below the solid solubility limit, and shall be especially applicable for UFG/ NC system.

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