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Effect of Addition of GO Nanoparticles on the Tensile Properties and Deformation Temperature of Sn-3.5Ag-0.7Cu Lead Free Solder Alloy

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The advantage of lead-liberated welding is the melting point of Sn-Ag-Cu (SAC) alloys in the Sn-rich alloy. Nanoparticles welding which requires lowering melting point near the Sn-Pb eutectic point is getting a growing interest. Recently, the phase persistence of nanoparticles has been the project of massiveness of academic and experiential investigations. In this study, graphene nanoparticles (GNPa) with 0.3 percentages wt. % were successfully added into Sn-3.5Ag-0.7Cu lead-free solder. The effects of graphene nanoparticles (GNPa) on the microstructure, tensile properties, wettability, corrosion resistance, and hardness were subsequently investigated. The results show that graphene nanoparticles (GNPa) refine the microstructure with different features and enhance the wettability efficiently. Stress-strain tests show that the combined solder containing 0.3 wt graphene nanoparticles (GNPa) exhibits about 15% and 25% enhancement in tensile strength and hardness, respectively. In addition, the total elongation of Sn-3.5Ag-0.7Cu is 22% greater than that of the pure Sn-3.5Ag-0.7Cu-0.3GO solder alloy. The enhancing mechanism of 0.3GO on the achievement of combined samples is also studied. Stress strain experiments were inspected under different five strain rates ranging from $5.4\text{x}10^{-5}$ S^{-1} to $2.9\text{x}10^{-3}$ S^{-1} and different five temperatures extended from R.T. (298K) to 383 K for two alloys. The activation enthalpy suggests that the dominant mechanism is the grain bounding diffusion (GBD). Also, X-ray diffraction examination display the permanence of both β-Sn rich phase and the intermetallic compound Ag_3Sn and very little particles or residue from the intermetallic composition γ-In Sn_4.

Keywords: Nanoparticles /Tensile properties / Free solder alloy

Introduction

The recent microelectronic industry, need solder; which are collective and plays an important function in suggestion electronic connection and mechanical backing for the precision of integrated circuits, which directly influences the realization of electronic application [1,2]. Sn-Ag-Cu alloys welder sample was excessively utilized through the latter days. The lead-free solder has been developed currently due to the harmful impact on the environment and human health [3-5].

Significant studies has been concentrated on the composition of Sn-Ag-Cu (SAC) [6-9] nanoparticles is appropriate and nominee for production of lead-free solders because of the lessening of melting degree. As a matter of reality, melting point depression in nanosolders was the main interest of most of these investigations, since the melting point of eutectic SAC bulk alloys is about 217 °C, about 34 °C higher than that of the of the conventional eutectic tin-37% lead alloy [10].

Many mechanisms such as the Gibbs-Thomson, has been before suggested to characterize the size reliance of the melting degree of nano-sized particles [11]. It is supposed that, as

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the melting is started by persistent vibrational lattice sensitivity on the solid surface, nanoparticles with considerable surfaces will miss their stability at reduced temperature than bulk. The melting point depression for Graphene Oxide (GO) nano may be qualified for instance in a conventional thermodynamic approximation by the Gibbs-Thomson equation [12], as follows:

$$T_m^{Nano}(r) = T_m^{bulk} - \frac{2(T_m^{bulk}+273.15)\sigma_s l}{\Delta H_f^{bulk}\rho_s r} \qquad (1)$$

Where σ_{sl} is the solid-liquid interfacial energy (~820 J/cm^2), ρ_s is the solid phase density of bulk alloy (7.39 g/cm^3) and, $\Delta H_f^{bulk}$is the latent heat of fusion of the bulk alloy (~ 67 J/g) for the eutectic SAC composition [13], T is fusion point. Other models prophesy a basically identical relationship as the Gibbs–Thomson equation, where T_m changes linearly with the reciprocal radius.

The CALPHAD (Computer Calculation of Phase Diagrams)-type thermodynamic characterization of nanoparticles samples is a massive contrivance for portend the phase diagram of nanoparticles about to or higher than 10 nm in diameter [14]. For the minimum particle size in the domain of little separated atoms, a bottom-up calculation such as molecular dynamics become true [15]. The CALPHAD-type for a nanoparticles sample process was first studied by Park and Lee [16].

For an identical melting system containing intermetallic particles like the Sn-Ag system, Sim and Lee [14] achieved latterly a regular survey, where the base Gibbs free energy of the stoichiometric compound (Ag_3Sn) was estimated based on the presence data for the surface pull of Ag_3Sn.

The ternary SAC eutectic alloy solder has different characteristic up the binary SA eutectic solder, due to its lower eutectic point, decelerate increase of the intermetallic category at the interface, excess strength, and minimize moistening angle [2,17]. Since the melting point of the ternary SAC alloys in the Sn-rich nook are of advantage for lead-free soldering, and many experimental researches on the phase deepness of SAC nanoparticles have been completed, the CALPHAD-type thermodynamic design of the binary SA nano-system has been elongated to the ternary SAC nano-system.

In order to improve the performance of Sn-3.5Ag-0.7Cu solder alloy, introducing minor of nanomaterials 0.3GO as reinforcing phases into the conventional solder alloy [2,18-20] is proved to be an effective and feasible method. Shen et. al. [21] reported that the nanoaddition could promote the hardening and creep resistance of alloys. Shen et. al., [22] found that the addition of nano refine and improve structure and mechanical properties. Yang et. al. [23] investigated the solder alloy enriched by Nano and point out the ductility and tensile strength were modified by bridging influence and pregnancy move. Graphene nanosheets (GNSs) are well known for its high thermal conductivity, excellent electronic properties and great mechanical strength [24-26]. Corresponding wettability and tensile strength improvement [27] in Sn-Ag-Cu solder alloy reinforced with the addition of Graphene nanosheets (GNSs) were obtained.

By the increment of Graphene nanosheets (GNSs), the abrasion resistance can be increased and the construction of inter metallic compounds (IMCs) enable as well be penned in the solder form during the consequence of Xu et. al; [28, 29]. In the study of Huetal [30], the increased hardness and the lower mounting rate of IMCs were gained by the strengthening techniques of the introduced grapheme in the Sn_8Zn_3Bi solder alloy. Maetal [31] found that the creep resistance [32]and ultimate tensile strength were enhanced significantly by addition of Graphene nanosheets (GNSs). Moreover, Pengand Deng [2,33] investigated the Graphene composite solder by finite element simulation and discovered ~192% enhancement in shear modulus with 1.0wt.% grapheme addition.

Experimental Procedures

The Graphene Oxide (GO) (99.99%purity) with an average particle size of 10 µm and density of 3.21 gm/cm^3 was used as powder substance; GO powders had a random morphology, it was fabricated by high energy ball milling technique, as described elsewhere [34, 35]. High purity GO powder was used as starting materials. The chemical compositions of two used solder alloys are Sn-3.5Ag-0.7Cu, and Sn-3.5Ag-0.7Cu-0.3GO, the Sn-3.5Ag-0.7Cu (SAC) solder was prepared from bulk Sn, Ag and Cu rods (all with 4 N purity), the Sn-3.5Ag-0.7Cu-0.3GO composite solders were prepared by mechanically mixing the GO nanoparticles with Sn-3.5Ag-0.7Cu-0.3GO alloy melt of the GO particles into the melt for 40 min to fixed a similar allocation of the growing of particles as seen in Table (1).

During preparation, the pre-weighted ingot (Sn-3.5Ag-0.7Cu-0.3GO) and GO particles were first put into an Al_2O_3 crucible and was completed in a vacuum arc furnace under the protection of high purity argon atmosphere at 450 ^{0}C for about 120 min. The blended particulates were pressed to predestine the sample with suitable dimensions. The powder particle was designed by increasing a little amount of powder in a resin followed by conventional grinding and polishing methods; to obtain an identical structure during the ingots, the samples was re-melted three times to product rod-like samples with a diameter of about 0.8 mm and 5 cm standard length. The sample was resigned at 25 ^{0}C for 168 hour before testing. More details of composite alloys preparation are described elsewhere [36-39]. This operation is declaration to permit a small amount of grain growth and grain stabilization to occur [40, 41]. The samples were tested at different five temperatures ranging from R.T. (298K) to 383 K for the two alloys under different strain rate ranging from 5.4x10^{-5} S^{-1} to 2.9x10^{-3} S^{-1}, using a conventional type creep machine [41]. The chemical compositions of the experimental alloys are represented in Table (1) and in Fig. (1); used Energy-dispersive X-ray spectroscopy (EDX) used in this investigation. The accuracy of temperature measurement is of the order ± 1 K. Strain measurements were done with an accuracy of ±1x10 $^{-4}$. A solution of 2% HCl, 3% HNO_3 and 95% (vol.%) ethyl alcohol was prepared and used to etch the samples.

Table (1): Chemical composition of the solder alloys studied(wt. %).

Solder alloy	GO	Sn	Ag	Cu
Sn-3.5Ag-0.7Cu	–	96.	3.5	0.7
Sn-3.5Ag-0.7Cu-	0.3	95.	3.5	0.7

Results and Discussion

Energy-dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD) of tested samples.

EDX, pattern for the tested alloys is symbolized in Fig. (1). Using EDX analysis, the percentage of Sn, Ag, and Cu are found similar to that value in Table (1). Fig. (2a) and b describe the XRD chart of the two solders. The acquired phases are characterized by draw an analogy between the Bragg peaks and the ASTM standards. A ponderous peak of β-Sn rich phase was constructed to be slightly diminutive with the extension of Cu, because of the construction of Cu_6Sn_5 phases aside from little peaks of Ag_3Sn. It is well recognized that, the disbanded Sn, Ag and Cu are condensed by presence of Ag_3Sn; the Ag_3Sn phase was found in the XRD pattern indicating the successful alloying of Sn and Ag after the melting point. The Cu_6Sn_5 IMCs in the tin matrix during solubility were owing to the mixing of Sn and Cupper [42,43]. Though, the intensity of GO composite is comparatively small, the major peaks of GO were exposed in Fig. (2b).

Microstructure change with addition of Ag, Cu, and GO

The SEM photograph are seen in Fig. (3a) Sn-3.5Ag-0.7Cu alloy microstructure composed of light grey areas of Ag_3Sn and dark network-like eutectic regions of β-Sn grain boundaries, and grey particles represented Cu_6Sn_5. In Fig. (3b), the suitable density of GO in the ternary solder was construct to upgrade the morphology, mechanical properties and are capable to refine the grain size, the Ag_3Sn IMCs, and the Cu_6Sn_5 perhaps doing as varied nucleation sites for β-Sn dendrites upon solidification and also white GO phase. It was explain that, the smooth in grain size originate from the existence of reinforcing GO samples which behave as pinners' to grain boundaries [37, 39]. This supervision may be characterized by the theory of diversified nucleation and the theory of adsorption of the surface active material. Therefore, the excess of adsorption of elements or oxides (GO) could decreasing the surface energy and reducing the dimension of IMCs of GO solder. Then, the least β-Sn grains as well; GO at the intermediate borders could be diffusion owing to the bounded solid solubility of GO in solder matrix. Thus, it is prophesy while combining GO is one of accepted nano-solder [44-45].

Mechanical properties

Tensile properties

The effect of GO Nanoparticles supplement on the mechanical properties of Sn-3.5Ag-0.7Cu samples at Room Temperature R.T. (298K), 333K, and 383 K and different strain rates were illustrated the relation of stress-strain curves as seen in Figs. (4-5) and Table (2). It was spotted that, the stress scales transferred across higher values with rising the strain rate and/or lessening the testing temperature. Significant of observation is that, the flow stress permanently lowered with growing elongation and plain solder alloy exhibited higher ductility than composite solder alloy. Usually, through plastic deformation solder alloys undergo simultaneous work hardening and dynamic recovery which have opposite effects on the plastic decreates additional dislocation forests during solder lattice space particularly at minimize testing temperature [38, 39]. Moreover, with increasing the testing temperature the dislocation annihilation takes place more quickly than dislocation propagation that is weakening the hardening. It is shown that Sn-3.5Ag-0.7Cu-0.3GO is more strengthening than Sn-3.5Ag-0.7Cu i.e. second alloys Sn-3.5Ag-0.7Cu are extra expansion than the GO Nanoparticles solders.

At constant strain rate equal $3.0x10^{-4}$ s^{-1} and different five testing temperature the effect GO additions of Sn-3.5Ag-0.7Cu; the tensile behavior were explained in Fig. (5) c,d. It is shown that the GO samples need more stress to reach the same strain, this mean GO samples are more in tensile strength by about 15%.

Table (2): Tensile testing condition

Solder alloy	Strain Rate (S^{-1})	Temp.(K)
Sn-3.5Ag-0.7Cu	5.4x10-5	298
	3x10-4	313
Sn-3.5Ag-0.7Cu-0.3GO	1.15x10-3	333
	2.9x10-3	358
		383

Influence of temperature and ε. Young's modulus (y), yield stress (σy), and σf.

Fig. (6a, b) lists the variation of Young's modulus (Y) with strain rate at different working temperature for Sn-3.5Ag-0.7Cu and Sn-3.5Ag-0.7Cu-0.3GO. It is clear that Young's modulus (Y) for composite samples is higher than that of plain samples. The Variation of yield stress (σy) with strain rate at different working temperature for Sn-3.5Ag-0.7Cu, and Sn-3.5Ag-0.7Cu-0.3GO is shown in Fig. (6-c,b); it is clear the yield stress (σy) for GO solders is more than that of plain samples. It is displayed that at the same testing temperature, raising the strain rate leads to altitude amounts of yield strength. The relation between the fracture strength (breaking strength) σf and strain rate at different working temperature for Sn-3.5Ag-0.7Cu-0.3GO and Sn-3.5Ag-0.7Cu is illustrated in Fig. (7a, b); but in Fig. (7c, d) the comparison the ultimate tensile stress (UTS) with strain rate for Sn-3.5Ag-0.7 Cu and Sn-3.5Ag-0.7Cu-0.3GO is represented. It is evident that fracture strength (σf) for GO nanosolders is more than that of other solders; the strain rate is directly proportional to fracture strength. The strain rate reliance of the total elongation ε_t was constructing to follow an experimental equation of the form [46]:

$$\varepsilon_t = A \exp(-\lambda \varepsilon^{\cdot}) \qquad (2)$$

Where A and λ are fixed constant depending on the tensile test situation

Strain rate and temperature dependence of the ultimate tensile stress (UTS).

Ultimate tensile stress (UTS) values of Sn-3.5Ag-0.7Cu, and Sn-3.5Ag-0.7Cu-0.3GOis inversely proportional with and temperature and directly with strain rate as demonstrated in Fig. (7 c, d). It is obvious that the value of (UTS) for nano samples is higher than that of ternary samples by about 11% as shown in Table (3 and 4). From Fig. (8-a, b); it is obvious that the total elongation in the two alloys is inversely proportional with strain rate and temperature at all strain rates; in Fig. (8a, b) Sn-3.5Ag-0.7Cu alloy has values of total elongation (ε_t) higher than that of GO particles by about 25%; due to softest nature of this alloy. This difference in the rapport between tensile elongation as characterization of strain rate and temperature appears to concern on the difference in the microstructure of the alloys. It is renowned that the tensile coefficient, the yield stress, fracture strength, and the total elongation of affected by the alteration of the strain rate and temperature as represented in table in Table (3, 4).

It is clear that SR (ε·) is directly proportional to the UTS in two tested samples; this is owing to rising strain rate is followed by rising in the dislocation density. As these dislocations shift they will complicated. It is then more complicated for else dislocations to elapse through the material, particularly at the minimized tested temperatures. The UTS value of GO nano sample presented is ultimate than Sn-3.5Ag-0.7Cu sample. The higher values of the UTS in the GO alloy may be due to the refinement and uniform distribution of the intermetallic Ag_3Sn, and Cu_6Sn_5 particles, it appear to supply a maximal measure of dispersion strengthening due to the finer particle size in the GO alloy compared to the Sn-3.5Ag-0.7 Cu alloy.

Table (3): strain rate on tensile parameters of Sn-3.5Ag-0.7Cu

Temp. K	Y	σ_y	σ_f	UTS	ε_f	ε_t%
298	3700	45	57	58	0.85	107
313	3520	41	48	48	0.79	102
333	3464	38	45	43	0.75	99
358	3210	31	36	35	0.73	83
383	3003	23	25	27	0.71	78

Table (4): strain rate on tensile parameters of Sn-3.5Ag-0.7Cu-0.3GO

Temp. K	Y	σ_y	σ_f	UTS	ε_f	ε_t%
298	4249	57	62	64	0.80	87
313	3930	44	53	55	0.76	75
333	3814	42	48	50	0.73	70
358	3654	36	41	41	0.70	62
383	3527	33	35	34	0.68	50

We can sight that, strain rate is inversely proportional with (ε_f) as ordinance in Fig. (8 c, d). It was celebrated that the yield stress (σ_y) and UTS are highly influenced by the distinction of strain rate (ε.), T and combination of GO nanoparticles. The value of σ_y and UTS is inversely comparative to the testing temperature and/or lowering the strain rate. These alterations can be known by regarding the plastic variations as thermally activated process and average conditioned of stress-assisted. Therefore, the dislocations have extra movement if thermal energy is raised and could dominate the barrier through β-Sn matrix [47, 48]. Hence, raising the temperature elevates the configuration of collection dislocation network into simple one. Therefore, at elevation strain rate, the σ_y and UTS are enhancement due to bounded time of the dislocation movement which produces heavy pinning locations and excess harden defects.

The reinforcement mechanism of the composite solder could be characterized in terms of harden GO nanoparticles and finer IMCs which spread within eutectic regions. They perform as defined centres which prevent the movement of dislocation that focused surrounded grain boundaries [49]. Furthermore, more disorder has been created with various slipping planes and various tendencies. Therefore, identical sign of consecutive dislocation would repel and collect in the domains of β-Sn grains. In extension to, the enormous slipping dislocations behave as tough obstructed web which is believe as one of the purpose for the strengthen mechanism of GO nanoparticles solder.

Zener pinning equation [50] was used to calculate the pinning stress that prevent the growth of β-Sn grains due to presence of finer IMCs and GO nanoparticles.

$$\sigma_{\mathrm{p}} = \frac{3\mathrm{f}\ \gamma_{GB}}{\mathrm{d}} \qquad (3)$$

where t is the particle volume fraction, d is the particle radius and γGB is the grain boundary energy of Sn ($\gamma_{GB}{}^{¼0.425}$ J/m^2) [51]. Thedefined stress valuesσp of Sn-3.5Ag-0.7Cu-0.3GO alloy are major than ternary solder alloy due to the presence of finer IMCs.

It is also interesting to note that the increased ductility of Sn-3.5Ag-0.7Cu than GO alloy is completed without sacrificing the mechanistic intensity. However, after the stress grade rise up to yield strength of the solder alloys, the strain hardening, instead of strain softening, has occurred in all the as-solidified solder alloys, which may consolidate the UTS and yield strength solders samples [52]. Since the distortion resistance of samples was defined crucial important mechanical property, a good deformation resistance suggests great plastic region. Compared with the Sn-3.5Ag-0.7Cu alloy and Sn-3.5Ag-0.7Cu-0.3GO alloy solders, which have a small misrepresentation resistance, a superior misrepresentation resistance and elongated ductile region of Sn-3.5Ag-0.7Cu alloy would insure this solder to become one of prefer for replacing the Sn-3.5Ag-0.7Cu-0.3GO solder in microelectronic packaging and interconnecting. The immovability to necking has been modified because of the high interface formed between Cu6Sn5 IMCs and the β-Sn matrix, which in turn enhanced both ultimate tensile stresses. While, the Ag3Sn IMCs particles formed in the Sn-3.5Ag-0.7Cu -0.3GO alloy can strongly enhance the ultimate tensile stress UTS of this alloy because of lower elastic modulus of these IMCs inside the solder matrix. These results are consistent with the previous finding that the Cu6Sn5 often exhibits a larger elastic modulus than Ag3Sn IMCs [52].

Stress exponents and activation energy

Tensile misrepresentation mechanisms are specified by the values of n and the activation energy Q. In fact, distortion of polycrystalline materials at temperatures over 0.5 Tm can happen by various deformation mechanisms, connected with various stress exponent amount. Propagation creep is related with n values nearly 1. The connection of stress-strain of Sn-based solder alloys is generally illustrated by the power-law of the kind [53-56]

$$\dot{\varepsilon} = A\,\sigma^n \exp(-Q/RT) \qquad (4)$$

The activation energy may be estimated from the connection between ln (fracture strain ε_f) with 1000/T at several working temperature for the two alloys as demonstrated in Fig. (9 a, b). The value of activation energy with temperature for solders is demonstrated in Fig. (9c).

Effect of temperature on stress exponent.

Fig. (10a) and b illustrated the relation between log (strain rate ε.) with log (σUTS) for both alloys at different temperatures. An evident linear relationship is spotted; this signifies that the relationship obeys [38];

$$\varepsilon = C\sigma^n \qquad (5)$$

Where σ is UTS, n is the stress exponent, and C is a constant for certain sample. The n values estimated from slope of two-logarithmic connection. Fig. (10c) abbreviated the gained n values for solders. These results illustrate that, then values decrease with increasing temperature; the value on n is around 2.39: 3.12 and from 3.44: 3.75 for two samples; respectively. Furthermore, the estimated of n quickly reducing for plain solder but remain comparatively stable for composite at fixed tested temperature. In fact; the distinction of n values with temperature is assigned to the rockiness of the microstructure of GO solder.

Mechanical alloying mechanism exemplifies to be a perfect way to synthesize nanocomposites in a different of systems. The ultimate apparent characteristic of the Mechanical alloying (MA) mechanism is that a symmetrical dispersion can be carried out by optimizing the processing contract. The elastic modules can be associated well with the additions of GO reinforcements in to the Sn matrix. The GO or (ZnO) advise to be favourable in that it gave good aggregation of slightly higher stiffer nature and fine particle size than Sn matrix, which was possible because of the reinforcement of hard GO or (ZnO) nanoparticles [57].

Conclusions

In this study, effect of GO nanoparticle weight percentage (0.3 wt %) additions on microstructure, thermal behaviour and tensile properties of Sn-3.5Ag-0.7Cu solder alloy have been investigated. The conclusions summarized as follows:

1. The GO extension reinforces the UTS and the YS while the ductility decreased for ternary alloys. That growing in UTS and YS can be known owing to dispersion encouragement influence of crucify and finer IMCs as well as solid solution crucify of GO content. In extension, the growing in UTS with elevate ε. can be demonstrated by dislocations reduplication and dislocation junction. Therefore, the supplement of GO can crucify the solder matrix released significantly variable the stress exponent or activation energies.
2. It is index to be a susceptible parameter and treatment parameters of the samples of the ductility variation were generally large for the ternary plain alloy than composite alloys.
3. The UTS values of raised with rising strain rate and lowered with rising temperature. The ternary alloy had the small values of UTS and more elongation, and composite alloy has contrary value.
4. The supplement of GO into the ternary samples can growing the solder distinguishing, like the ultimate tensile strength (UTS), and diminish ductility. This is inasmuch the presence of intermetallic combination (IMCs) Cu_3Sn and Ag_3Sn
5. The tensile parameters, the yield stress and the total elongation are highly influenced by temperature strain rate and.

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