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Young's modulus and creep compliance of GaAs and $Ga_{1-x}Mn_xAs$ ferromagnetic thin films under thermal stress at varied manganese doping levels

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Dynamical mechanical analysis yields information about the mechanical properties of a material as a function of deforming factors, such as temperature, oscillating stress and strain amplitudes. GaAs and Mn-doped GaAs at varied levels, used in making electronic devices, suffer from damage due to changes in environmental temperatures. This is a defective factor experienced during winter and summer seasons. Hence, there was a need to establish the best amount of manganese to be doped in GaAs so as to obtain a mechanically stable spin injector material to make electronic devices. Mechanical properties of $Ga_{1-x}Mn_xAs$ spin injector were studied in relation to temperatures above room temperature (25 °C). Here, creep compliance, Young's moduli and creep recovery for all studied samples with different manganese doping levels (MDLs) were determined using DMA 2980 Instrument from TA instruments Inc. The study was conducted using displace-recover programme on DMA creep mode with a single cantilever clamp. The samples were prepared using RF sputtering techniques. From the creep compliance study it was found that MDL of 10 % was appropriate at 30 °C and 40 °C. The data obtained can be useful to the spintronic and electronic device engineers in designing the appropriate devices to use at 30 °C and above or equal to 40 °C.

Keywords: *creep compliance; Young's modulus; percentage creep recovery; strain jumps; manganese doping levels*

1. Introduction

1.1. GaAs and $Ga_{1-x}Mn_xAs$ thin films

GaAs films are prepared by direct reactions of the elements of gallium and arsenic in three similar industrial processes, namely:

1. vapour phase epitaxy (VPE) reaction of gaseous gallium metal and arsenic trichloride as shown in the chemical equation:

$$2Ga + 2AsCl_3 \longrightarrow 2GaAs + 3Cl_2 \quad (1)$$

2. metal organic chemical vapour deposition (MOCVD) reaction of trimethylgallium and arsine as shown in the chemical equation:

$$Ga(CH_3)_3 + AsH_3 \longrightarrow GaAs + 3CH_4 \quad (2)$$

3. molecular beam epitaxy (MBE) of gallium and arsenic as represented by chemical equation:

$$4Ga + As_4 \longrightarrow 4GaAs \text{ or } 2Ga + As_2 \longrightarrow 2GaAs \quad (3)$$

GaAs has a direct band gap and can be used to manufacture devices such as microwave frequency integrated circuits, monolithic microwave integrated circuits, infrared light emitting diodes, laser diodes and solar cells. The band gap, electron mobility and electronic conductivity of GaAs can be modified by addition of impurity atoms of

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manganese to form $Ga_{1-x}Mn_xAs$ for nanoelectronics or nanotechnology. The increase in manganese doping level monotonically increases the electron mobility and electron conductivity, hence, the data computing speed [1].

The $Ga_{1-x}Mn_xAs$ is an anisotropic material consisting of three elements, combined chemically by molecular beam epitaxy (MBE) or magnetron sputtering technique. Manganese atoms are added to the GaAs host substrate as impurities that create conduction charge carriers with magnetic spin moment. The doped manganese substitutes for Ga site in the host semiconductor, GaAs, to produce $Ga_{1-x}Mn_xAs$.

1.2. Creep recovery testing

Deformation of a material occurs when a load (force) is applied to it and it deforms plastically under an applied stress depending on the strength of intermolecular forces [2, 3]. After an initial deformation, the deformation of the material later reaches its maximum. The deformation can then be plotted against time and temperature [4, 5]. More precisely, representative samples of materials can be tested for creep. The sample is loaded with a very low stress level just enough to hold it in place and allow it to stabilize. The testing stress is then applied very quickly, with instantaneous application, and changes in the material response are recorded as percent strain [6, 7]. The material is then held at this stress for a period of time until it reaches equilibrium. Creep tests can be used in two ways: to obtain fundamental information about a material or to investigate the material response under the real use conditions [8, 9].

1.3. Creep - recovery analysis

Creep-recovery data results can be interpreted in three ways: a plot of strain versus stress can be done and data fitted to the model (four element model) or a plot of strain versus stress and quantitative analysis done in terms of irrecoverable creep, viscosity, modulus and relaxation time [10]. Lastly, a plot of creep compliance versus time (temperature) can be drawn. The creep compliance is a mechanical property that measures the tendency of a material to respond to deformation and confirms material softness above glass transition temperature (T_g) [9]. As stated earlier, there is an immidiate response of a material to an applied stress (load) and the point at which the stress is applied is assumed to be the starting point (time zero) for the creep experiment. For the recovery portion, the time zero point is when the stress is removed. The initial jump is equivalent to the applied stress divided by a spring constant ($\frac{\sigma}{E_1}$) which is envisioned as an immediate stretching and locking into its extended condition. Practically, this region is very small and could be difficult to be seen and the strain time derivative may be used to locate it. After the spring is extended, the independent dashpot and the Voigt element can respond.

When the load is removed there is an immediate recovery of this spring, equivalent to $\frac{\sigma}{E_1}$ [11, 12]. This can be observed as an elastic deformation of material chains. The slope of the straight equilibrium region of the creep curve gives the strain rate. Initial and recoverable strains can be determined, and in the region of a constant strain rate, equillibrium viscosity (η_C) can be obtained [4, 13, 14]. Percent recovery can also be calculated to provide information on how much of a material regains its original properties (resilience) after the stress (load) is released. Recovery time provides the amount of time required for strain to recover to 36.79 % of its original value [16]. A plot of creep compliance versus temperature can be analysed to observe, where the properties degrade as temperature increases. Temperature can be raised and lowered so as to simulate the effect of an environmental thermal cycle [17]. When the stress on a sample is increased, the material may creep under the applied load and when the load is removed the sample may attempt to recover its original dimensions (property of stress relaxation) [11, 12].

GaAs and manganese doped GaAs used in making electronic devices endure damage caused by adverse environmental temperatures. This causes mechanical instability of the associated devices which is a major problem to spintronic device engineers. Hence, there was a need to determine the appropriate amount of manganese that can be doped in GaAs to achieve a mechanically stable spin injector material that can withstand harsh environmental temperature conditions.

2. GaAs and $Ga_{1-x}Mn_xAs$ ferromagnetic sample preparation

The $Ga_{1-x}Mn_xAs$ films were deposited by radio frequency (RF) magnetron sputtering technique in the Laboratory of Semiconductor Films, São Paulo State University (Bauru/Brazil), courtesy of Advanced Materials Group. The films were grown in an Ar atmosphere (99.9999 % purity). A 100 mm diameter, 6 mm thick, electronic grade undoped GaAs wafer (Ramet Technology) was used as a target in a planar diode configuration. The amorphous silica (a-SiO_2) substrate was fixed directly opposite to the target at a distance of 50 mm. Manganese was added to the films by a co-sputtering process; that is, pure Mn slabs (99.99 %) were placed onto the GaAs target which was also subjected to the sputtering process, resulting in incorporation of Mn into the films. The concentration of Mn incorporated into the films was controlled by covering different fractions of the target area with Mn slabs. The thin films of a thickness of 500 to 1000 nm were then removed from the chamber and cut into rectangular samples of the length of 12.96 mm and width of 0.99 mm.

2.1. Experimental

The prepared samples, due to their short lengths, were designed to fit the clamp by mounting them on long microscope slides of the same dimensions as the standard steel sample at their centres, as shown in Fig. 1. A control sample was also prepared to correct the error caused by microscope slides by mounting SiO_2 microscope slides of the same thickness and lengths as the GaAs and $Ga_{1-x}Mn_xAs$ as shown in Fig. 2.

DMA 2980 instrument from TA instruments was used to carry out the tensile tests. The single cantilever clamp was used because of rectangular shape of the samples of dimensions 39.81 mm × 12.7 mm × 0.99 mm. The DMA creep mode was also used on a displace-recover programme of 10 minutes: 10 minutes, respectively, at 30 °C and 40 °C. The same procedure was used for all studied samples and the actual tensile parameter values were obtained by calculating the deviations (shifts) from the values of undoped GaAs, recorded in Tables 1 and 2. The samples were clamped as shown in Fig. 3.

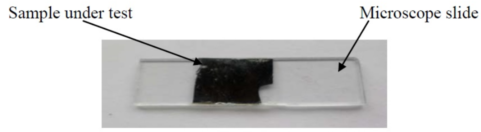

Fig. 1. Study sample mounted on a microscope slide.

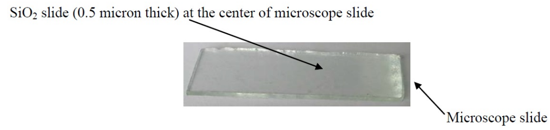

Fig. 2. SiO_2 mounted on a microscope slide at the center.

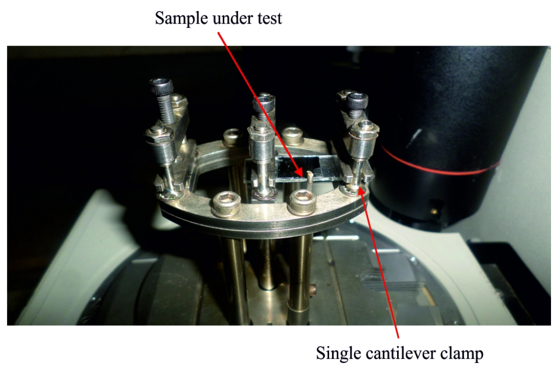

Fig. 3. Modified study sample clamped with a single cantilever.

2.1.1. Computational theory

The creep compliance and Young's moduli are crucial parameters in this study as they represent the stiffness of the material and furthermore explain how a material response to all sorts of deformation due to changes in stress amplitude, strain amplitude and temperature. The thermal stress-thermal strain relationships for GaAs and $Ga_{1-x}Mn_xAs$ were plotted by simulation of the data obtained with the DMA 2980 using the TA universal analysis software. Similar process was followed for the case of creep compliance. The Young's moduli were calculated from the slopes of the linear regions of thermal stress-thermal strain graphs at 30 °C and 40 °C and tabulated as functions of MDLs. 30 °C was used as a working temperature during the study to represent the average room temperature. It was also necessary to study the mechanical stability situation of the thin films at an elevated temperature. Therefore, 40 °C was also used because it was assumed that the mechanical properties of the thin films under study between 30 °C and 40 °C will provide useful information.

3. Results and discussion

3.1. Creep compliance at 30 °C

Fig. 4 shows creep compliance: MDLs relationships at 30 °C and 40 °C plotted from the data in Table 1. From the two data sets, it is apparent that creep compliance is in an increasing trend with the MDLs. Pure GaAs has the smallest creep compliance value of 0.0604 $\mu m^2/N$ and GaAs doped with 50 % manganese atoms shows the highest value of 0.7864 $\mu m^2/N$. The creep compliance for MDLs of 1 %, 10 % and 20 % are 0.0996 $\mu m^2/N$, 0.4704 $\mu m^2/N$ and 0.5614 $\mu m^2/N$, respectively. Inferring from the gradients of the graphs plotted for MDLs of 0 % to 10 %, the creep compliance increases monotonically. However, beyond 10 %, the creep compliance increases gently. This means that the manganese impurity atoms have a significant influence on the creep compliance of the dilute magnetic semiconductor. It is believed to cause a profound alteration of the GaAs crystal structure that makes it porous and softer. This is because the atoms have enough space to accommodate the changes resulting from deformation [9]. The imperfections beyond MDL of 10 % are minimal. So, generally, at 30 °C, the $Ga_{1-x}Mn_xAs$ with MDLs of 20 % to 50 % provides the best results because of their respective high creep compliance compared to the other samples.

Intrinsically, the mechanical behaviour of a material is closely related to its structure, including bond strength. However, the mechanical behaviour of a crystalline material is also controlled by imperfections, such as vacancies and interstitials [18, 19]. Therefore, here, the higher creep compliance implies that the material is not on the verge of breaking as it responds well to the imperfections and deformations. At elevated temperature of 40 °C, the responses of creep compliances to the variations in MDLs are significantly similar to those at 30 °C. This can be observed in Fig. 4 plotted from the data in Table 1.

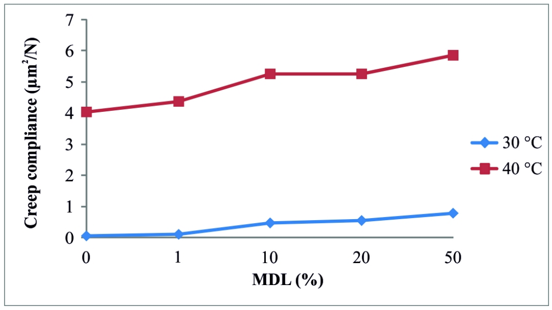

Fig. 4. Creep compliance versus MDL at 30 °C and 40 °C.

Table 1. Creep compliance at different MDLs.

MDLs	Creep compliance ($\mu m^2/N$) 30 °C	Creep compliance ($\mu m^2/N$) 40 °C
0 %	0.0604	4.0394
1 %	0.0996	4.3750
10 %	0.4704	5.2501
20 %	0.5614	5.2530
50 %	0.7864	5.8512

3.2. Creep compliance at 40 °C

From Fig. 4 and Table 1, it can be seen that MDL of 10 % reveals the highest creep compliance of 5.2501 $\mu m^2/N$ which is of 29.9723 % higher in comparison to pure GaAs with creep compliance of 4.0394 $\mu m^2/N$. MDLs of 0 % and 1 % show the least creep compliance of 4.0394 $\mu m^2/N$ and 4.3750 $\mu m^2/N$ at 40 °C.

At these MDLs, the $Ga_{1-x}Mn_xAs$ samples are on the verge of breaking as they do not respond easily to the temperature deformation. This is attributed to the perceived reduced interatomic spacing (free volume) that reduces allowance for expansion or molecular movements upon deformation [12, 20]. At MDL of 50 %, the manganese underfill becomes saturated as it is perceived to create excess atoms with more space for interatomic movements. Hence, the creep compliance increases to 5.8512 μm^2/N. Beyond MDL of 20 % manganese atoms create extra internal movements as a result of enough interatomic space that is in increasing trend. Therefore, MDL of 50 % has more molecular motions than other MDLs, which is expressed by the high creep compliance of 5.8512 μm^2/N. This implies that at 40 °C, the increasing levels of MDLs gradually soften the GaAs crystal lattice by creating more interatomic spacing that allows the material to respond well to the deformation and avoid sudden breakages.

Generally, the noticeable effect of temperature on the creep compliance was observed. By considering Fig. 4, it can be seen that at MDL of 0 %, the creep compliance is 0.0604 μm^2/N at 30 °C and 4.0394 μm^2/N at 40 °C. This shows that the crystalline structure of GaAs becomes more disordered at temperature of 40 °C compared to the structure at lower temperature of 30 °C. This implies that more free volumes are created so that any aspect of deformation causes excessive molecular motion. This is consistent with a sharp increase in creep compliance to 4.0394 μm^2/N.

3.3. Young's modulus for MDLs samples at 30 °C

Young's modulus is a measure of mechanical strength of a material, given as a ratio of stress to strain. This mechanical parameter gives information about the tendency of a material to resist deformative forces. In Fig. 5, Young's modulus vs. MDL has been drawn using the data from Table 2 at 30 °C. The Young's modulus drops to 3.2870 × 10^7 Pa for MDL of 1 % from 4.1873 × 10^7 Pa for MDL of 0 %, then it further decreases to 3.1375 × 10^7 Pa for MDL of 10 %. Between 10 % and 50 % MDLs, the Young's modulus increases gently up to 3.2470 × 10^7 Pa for MDL of 50 %. The addition of manganese impurity into the pure GaAs up to 1 % causes an irregular increase in thermal strain and a reduction in thermal stress. The reduction in thermal stress implies that the cross sectional area increases, while the thermal force is relatively constant. Temperature of 30 °C is sufficient to generate an increased mechanical strength up to 3.2430 × 10^7 Pa for MDL of 50 %.

Table 2. Young's moduli at different MDLs.

MDLs	Young's modulus (×10^7 Pa)	
	30 °C	40 °C
0 %	4.1875	3.5331
1 %	3.2870	2.7970
10 %	3.1375	2.9196
20 %	3.1745	3.0062
50 %	3.2430	3.0106

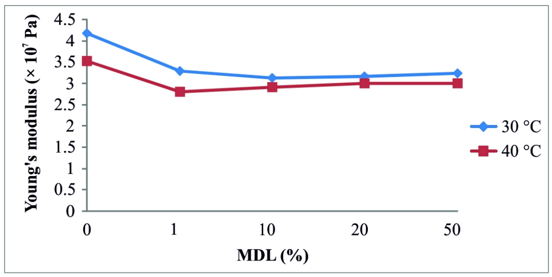

Fig. 5. Young's modulus versus MDL at 30 °C and 40 °C.

3.4. Young's modulus for MDLs samples at 40 °C

In Fig. 5, Young's modulus vs. MDL has been plotted using the data from Table 2 at 40 °C. It can be observed that the change in mechanical strength with MDLs takes a similar pattern as in 30 °C. Pure GaAs seems to be the strongest, with Young's modulus of 3.5331 × 10^7 Pa which then drops drastically upon addition of 1 % MDLs to 2.797 × 10^7 Pa. For MDLs between 10 % and 50 %, Young's modulus increases monotonically up to 3.0106 × 10^7 Pa. This shows that the Mn-doped GaAs at MDL of 1 % and 10 % are not strong enough to resist the breakage due to thermal strain forces at 40 °C.

3.5. Creep recovery and initial deformation analysis at 30 °C

According to Fig. 6 and Table 3, the initial strain jump at 30 °C generally decreases with an increase in MDL. At MDL of 1 %, there is an increase in strain jump from 1.6578×10^{-4} % for pure GaAs to 1.7368×10^{-4} %. This implies that the applied stress, in case of manganese dopant of 1 %, creates more space (free volume) for internal molecular motions (chain slippage) and the material becomes softer. For the range of MDL from 1 to 10 %, the manganese atoms occupy intermolecular spaces and reduce the space that would have been available for internal movements [20, 24–26]. This makes the material rigid. With further increase in MDL, the strain decreases gradually and tends to the value of 1.1569×10^{-4} % at MDL of 50 %. In Fig. 7 and Table 4 at 30 °C, it can be seen that the material recovers upon withdrawal of stress at MDL of 1 % with 100.00 % creep recovery. This shows that at 30 °C, the GaAs doped with 1 % manganese becomes disordered but upon withdrawal of stress it recovers fully to its original dimension. This implies that the material becomes softer (more compliant) and plastic.

Table 3. Initial strain jump at different MDLs.

MDLs	Initial strain jump ($\times 10^{-4}$%)	
	30 °C	40 °C
0 %	1.6578	3.9737
1 %	1.7368	3.375
10 %	1.3026	3.5625
20 %	1.2763	3.3125
50 %	1.1569	3.1875

The trend persists (at 100 %) up to MDL of 50 %. This shows that at MDL beyond 1 % there is no loss of stored energy through thermodegradation at 30 °C.

3.6. Creep recovery and initial deformation analysis at 40 °C

At 40 °C, according to Fig. 6 and Table 3, MDL of 1 % causes a reduction in the initial strain jump which is contrary to the situation at 30 °C. This suggests a reduced free volume for chain slippage [14]. So, a 10 °C temperature increase suppresses the manganese of 1 % and causes a decrease of creep percentage recovery to 97.42 % as shown in Table 4 and Fig. 7. Beyond MDL of 1 %, the creep recovery almost plateaus up to 97.43 % at MDL of 50 %. The general reduction in creep percentage recovery at 40 °C implies that a lot of stored energy is being dissipated by internal friction as a result of chain slippage which has been restricted by a reduced free volume [20–23]. This makes the Mn-doped GaAs more rigid. The initial reduction in the initial deformation to 3.375×10^{-4} % at MDL of 1 % is followed by a drastic increase up to 3.5625×10^{-4} % at MDL of 10 %. Beyond MDL of 10 %, initial strain jump generally drops to 3.1875×10^{-4} % at MDL of 50 %. This can be observed in Fig. 6 and Table 3 which provide a similar pattern as the observations made at 30 °C.

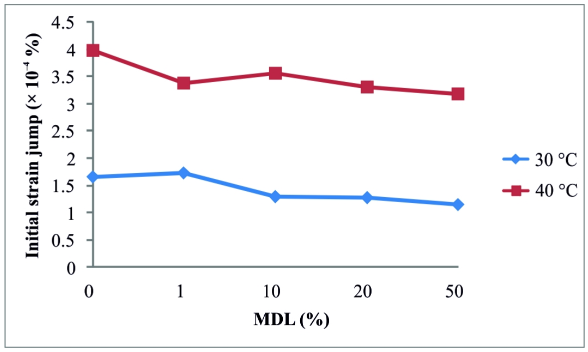

Fig. 6. Initial strain jump versus MDL at 30 °C and 40 °C.

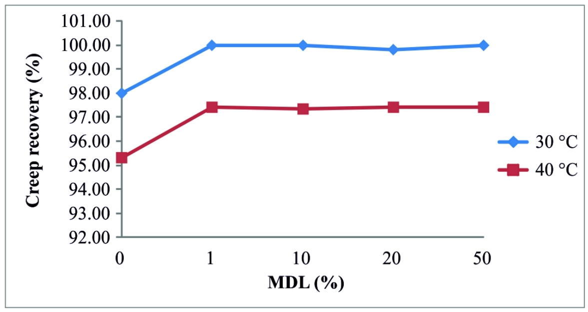

Fig. 7. Creep recovery versus MDL at 30 °C and 40 °C.

Generally, the two different observations at 30 °C and 40 °C show that temperature variation has an impact on the deformation stress compliance of Mn-doped GaAs. Temperature variation does not have a significant effect on the creep percentage recovery as shown in Fig. 7. In Fig. 7 the trend of the variation of creep percentage recovery with MDLs at 30 °C and 40 °C is similar. The effect of deformation stress that causes varying initial deformation is purely a function of temperature and it is responsible for increasing and reducing free volumes at different MDLs.

Table 4. Creep recovery at different MDL.

MDLs	Creep recovery	
	30 °C	40 °C
0 %	97.98 %	95.33 %
1 %	100.00 %	97.42 %
10 %	100.00 %	97.33 %
20 %	99.82 %	97.40 %
50 %	100.00 %	97.43 %

It is also apparent that MDLs have an effect on deformation stress at a given temperature. The presence of lattice defects also influences the mechanical strength. A qualitative explanation for the composition dependence of hardness can be given in terms of two contributions. One is the lattice contribution and another one is the presence of defects, such as vacancies, impurity-vacancy pairs and dislocations [27, 28]. In spintronic device designing the material to use ought to recover fully from deformation stresses and increase in free volume (softening) at relatively high temperature. This is to appropriately operate in hotter regions of the world and increase the mechanical stability of such devices. The increased mechanical stability has significant influence on the fabrication and processing of spintronic devices. It makes the devices easy to polish with less cracking [29].

4. Conclusions

Electronic devices, such as transistors made from GaAs and $Ga_{1-x}Mn_xAs$, should be used at environmental temperatures of approximately 30 °C and with MDLs of between 10 % and 50 %. This is because the $Ga_{1-x}Mn_xAs$ spin injector is not in any danger of breakage due to its high creep compliance of 0.4704 $\mu m^2/N$ at 30 °C and 5.2501 $\mu m^2/N$ at 40 °C and high creep percentage recovery of 100 % at 30 °C and 97.33 % at 40 °C. Thus, it can stretch and recover without breaking and without creating an obstruction to the flow of spin charge carriers responsible for the conduction of the spin current. The electronic devices made of $Ga_{1-x}Mn_xAs$ are not suitable for the use at environmental temperatures of approximately 40 °C and above as they have shown to be defective. This is because there is a danger of breakage of the spin injector which then cuts the path for the flow of the spin current. At elevated temperatures (40 °C), pure GaAs is stiff to bow to any strain deforming forces, which is shown by 97.98 % creep recovery at 30 °C and 95.33 % at 40 °C.

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