Thin films consisting of hydrogenated amorphous silicon (a-Si:H), in which germanium nanocrystals (nc-Ge) are embedded, have been synthesized using a dual-chamber co-deposition system. The thermopower and conductivity are studied as the germanium crystal fraction $X_{\text{Ge}}$ is systematically increased. For $X_{\text{Ge}} < 10\%$, the thermopower is n-type (as found in undoped a-Si:H), while for $X_{\text{Ge}} > 25\%$ p-type transport is observed. For films with $10\% < X_{\text{Ge}} < 25\%$, the thermopower shifts from n-type to p-type as the temperature is lowered from 450 K to 350 K. The n-type to p-type transition is sharper than expected from a standard two-channel parallel conduction model for charge transport. 

I. INTRODUCTION

There has been growing interest in the properties of complex materials that show order on length scales intermediate between the atomic and macroscopic. Examples of mesoscale materials include composite nanostructures and thin films of an amorphous matrix containing nanocrystalline inclusions. These complex systems can display novel electronic transport characteristics that transcend either system alone. The low-dimensionality and short length scales of nanocrystals can have a profound influence on a semiconductor’s properties, including the energy gap. This provides a new degree of freedom, in addition to chemical composition and atomic bonding arrangements, with which the electronic properties of semiconductors can be varied and controlled.

For example, the conductivity of composite films consisting of undoped hydrogenated amorphous silicon (a-Si:H) containing silicon nanocrystals (nc-Si) of diameter $\sim 5-6 \text{ nm}$ is very sensitive to the crystal volume fraction, with an enhancement of the dark conductivity of several orders of magnitude for crystal fractions of $\sim 2\%-4\%$. This has been ascribed to a modulation doping effect, where thermally excited charges in the nc-Si are transferred to the surrounding a-Si:H matrix. Unlike in the defect equilibration model of doping in amorphous semiconductors, the midgap defect density for these low crystal fraction films is unchanged compared to a-Si:H without nanocrystal inclusions. Mixed-phase films with higher crystal fractions exhibit a lower conductivity than when the crystal fraction is $\sim 2\%-4\%$, though still above that found in pure a-Si:H. This reduction in conductivity for higher crystal fractions is attributed to a higher defect density in these films, pulling the Fermi energy back towards the middle of the bandgap.

In n-type doped mixed phase films of a/nc-Si:H, the conductance is complex and three distinct charge transport mechanisms are observed: thermally activated transport through the a-Si:H; multi-phonon assisted hopping through the nanocrystals; and Mott-Variable Range Hopping through bandtail states, as the crystal fraction and temperature are varied.

Thin films of a-Si:H are typically deposited via plasma-enhanced chemical vapor deposition (PECVD), and by adjusting the deposition conditions one can introduce nanocrystalline inclusions into the amorphous semiconductor. The recent construction of a dual-chamber co-deposition system, in which nanocrystalline particles are synthesized in an upstream flow-through tube plasma reactor and are then injected into a second capacitively-coupled plasma (CCP) deposition system, enables the synthesis of mixed-phase materials of a nanocrystalline phase A in a thin film matrix B. It is therefore possible to extend these studies of transport phenomena of nc-Si in a-Si:H to other semiconducting nanocrystals embedded within a-Si:H films.

We report here the synthesis and electronic properties of mixed-phase thin films of hydrogenated amorphous silicon (a-Si:H) in which nanocrystalline germanium (nc-Ge) particles are homogeneously embedded. The electrical properties of nc-Ge/a-Si:H composites are studied as the germanium crystal fraction, $X_{\text{Ge}}$, is systematically varied from 0% to 75%. The sign of the majority carrier, as reflected in measurements of the Seebeck coefficient, changes as the germanium crystal fraction is increased. Transport is found to be through the a-Si:H matrix for small germanium crystal fractions ($X_{\text{Ge}} < 10\%$), and through the nc-Ge phase for $X_{\text{Ge}} > 25\%$. Experimental studies of electronic conductivity during a percolation transition typically focus on binary systems, where a conducting material is interspersed within an insulating matrix, for example, metallic gold particles or silicon nanocrystals embedded within a silicon oxide film. The high resistivity contrast of the two materials facilitates the determination of when a continuous pathway of conducting particles has formed as the concentration of metallic particles increases, but at the expense of information concerning the sub-threshold state. In the nc-Ge/a-Si:H material system, the transition from conduction through the a-Si:H matrix to through the nc-Ge phase is reflected in measurements of the thermopower, which finds a change from n-type (for...
$X_{\text{Ge}} < 10\%$) to p-type transport (for $X_{\text{Ge}} > 25\%$) as the nc-Ge concentration is increased. For germanium crystal fractions in the intermediate range ($10 < X_{\text{Ge}} < 25\%$), the thermopower is p-type near room temperature and n-type above $\sim 400 \text{K}$.

This paper is organized as follows. The dual-chamber co-deposition system and growth conditions for these films are described. Structural characterizations, using X-ray diffraction, Rutherford Back Scattering, and Raman spectroscopy, of the nanocrystal diameter and density are then reported. Thermopower and conductivity data are presented as the germanium content is varied for these films. These results are then analyzed in the context of a simple two-channel model of charge transport. Calculations of the expected variation in the thermopower as a function of nc-Ge concentration are found to qualitatively describe the p- to n-type transition, but are quantitatively inconsistent with the entire observed transition. A preliminary report of the properties of these films has been published separately.\(^{12}\)

II. MATERIALS PREPARATION

The nc-Ge/a-Si:H films described here were grown in a dual-chamber co-deposition PECVD system,\(^{8}\) sketched in Fig. 1. A mixture of germane (GeH$_4$) and argon flow through an upstream nanoparticle synthesis chamber,\(^{13}\) which consists of $3/8\text{''}$ quartz tube with ring electrodes, for which the deposition conditions (100 W, 4.5 Torr) are optimized to promote the growth of monodisperse germanium nanocrystals. The nc-Ge are then entrained by the argon flow and injected into the second CCP reactor, in which a-Si:H is deposited from silane in a plasma at a lower power and pressure (4 W, 0.25 Torr). The synthesis of the nanocrystals is completely decoupled from the deposition of the host semiconductor matrix, so that the growth conditions can be independently optimized for each component of the composite material. The co-deposition system had recently been completely rebuilt prior to the synthesis of these nc-Ge/a-Si:H films and neither deposition chamber has been exposed to either phosphine or diborane. Consequently, unintentional doping by contaminant gases is excluded as an explanation for the results described here.

The typical film thickness is $\sim 1 \text{\mu m}$, and the films are deposited onto Corning 1737F quartz at a substrate temperature of $250 \text{\degree C}$. Aluminum electrodes (thickness $\sim 100 \text{nm}$) are deposited in a co-planar configuration onto the surface of the films as shown in Fig. 2. Linear current-voltage characteristics are observed for all of the films and at all temperatures presented here. Owing to gas convection in the CCP chamber, the concentration of nc-Ge embedded in the a-Si:H decreases as a function of distance from the injection tube. We are therefore able to grow a series of nc-Ge/a-Si:H films in a single deposition run, where only the nc-Ge concentration is varied. These are ideal samples for investigating the influence of nanocrystal density on the electronic properties of the mixed-phase films, as both the nc-Ge diameter and surrounding a-Si:H matrix are identical within a given deposition series, and the only parameter varied is the concentration of nc-Ge. Following deposition of aluminum electrodes, the film is partitioned using a diamond scribe so that up to four separate samples, each of width 2.5 mm, can be measured. Each deposition run yields at most ten samples of varying crystal content. The average diameter of the germanium nanocrystals varies slightly from run to run from 3.5 to 4.5 nm, depending on the specific deposition parameters employed in the top nanoparticle synthesis chamber. However, the results presented here are not sensitive to this minor diameter variation, and data from several such deposition runs are compared.

III. STRUCTURAL CHARACTERIZATION

The presence of nc-Ge within the a-Si:H matrix is confirmed using X-ray diffraction (XRD) and Raman spectroscopy. The XRD measurements were performed using a Bruker-AXS Microdiffractometer with a 2.2 kW sealed Cu X-ray source ($\lambda = 1.5418 \text{\AA}$). A sample XRD curve with the background and amorphous silicon signals subtracted is shown in Fig. 3. The widths of the diffraction lines are used
to extract the average diameter of the nanocrystals responsible for the scattering peaks. A separate data set collected using a Corundum sample (Al₂O₃), with a negligible natural linewidth compared to instrumental factors, was used to calibrate the inherent instrumental broadening. Analysis of X-ray diffraction peak widths using the Scherrer equation finds that the germanium nanocrystals in our films have diameters between ~3.5 and 4.5 nm.

The relative concentration of nc-Ge compared to a-Si:H in these composite films was quantified using Raman spectroscopy and Rutherford Backscattering (RBS). As described above, a typical mixed-phase film deposited on a glass substrate will exhibit a gradient of germanium concentration along the length of the film. After the deposition of electrodes and scribing the film into four distinct samples, the absolute germanium concentration was determined using Rutherford Backscattering. For the film sketched in Fig. 2, the germanium volume fractions for the individual samples labeled 1, 2, 3, and 4 are 0.3%, 1.8%, 7.5%, and 18%, respectively, as determined by RBS.

Raman spectra were recorded with a 514.5 nm argon ion laser at a power of less than 6 mW with care taken to avoid heating of the films. Representative Raman spectra are shown in Fig. 4 for samples grown in a single deposition run, with some films being omitted simply for clarity. The sharp small size of the Si-Ge peak at 390 cm⁻¹ broadens the peak near 390 cm⁻¹ shown in Fig. 4 for samples grown in a single deposition run, local heating of the films. Representative Raman spectra are recorded with a 514.5 nm argon ion laser at a power of less than 6 mW with care taken to avoid heating of the films. Representative Raman spectra are shown in Fig. 4 for samples grown in a single deposition run, with some films being omitted simply for clarity. The sharp feature at 300 cm⁻¹ is consistent with the c-Ge TO mode, while the broad peak at 480 cm⁻¹ is associated with a TO excitation of the amorphous silicon matrix. The broad feature near 390 cm⁻¹ has been ascribed to Si-Ge bonds in studies of homogenous a-GeₓSi₁₋ₓ:H alloys. The small size of the Si-Ge peak at 390 cm⁻¹ compared to the a-Si:H and nc-Ge TO modes suggests that the material is largely phase-separated, and there is very little homogeneous Si-Ge alloying. The germanium crystal fraction X_{Ge} for each sample presented here was determined from the fractional integrated area of the nc-Ge peak at 300 cm⁻¹ compared to the a-Si:H TO mode at 480 cm⁻¹. Knowing the total germanium content from RBS for representative films enables us to ascertain the size of the nanocrystals

IV. ELECTRONIC TRANSPORT PROPERTIES

As the nc-Ge concentration in these films is increased, charge transport changes from n-type to p-type, as reflected in measurements of the temperature dependence of the thermoelectric coefficient (the Seebeck effect). In this measurement, the sample rests across two separate copper blocks (H1 and H2 in Fig. 2); inside of each Cu block, a 50 W cartridge heater is embedded. The temperature of each block is controlled independently by a dual-channel temperature controller. The temperatures of the blocks T₁ and T₂ are set so that the average is T_{avg} = (T₁ + T₂)/2 and the temperature gradient ΔT = T₂ - T₁ induces a thermoelectric voltage (V₁–V₂ in Fig. 2) across the sample. For each average temperature, we generate thermal gradients of ΔT = ± 12°C and 0 and record the respective induced voltage, eliminating contributions of any small temperature-dependent voltage offset to the signal. Temperature stability is maintained within ±0.05 K of the target set temperature for at least 90 s before collecting data. The entire measurement system is capable of measuring the electrical properties of high impedance thin films down to σ ~ 10⁻⁵ Ω⁻¹ cm⁻¹ and resides in a vacuum chamber. The measured Seebeck coefficient is derived from the slope of the resulting linear plot of induced voltage against ΔT, and the process is repeated at a new average temperature, from 350 K to 450 K, as shown in Fig. 5. Fig. 5(a) shows a series of plots for a single composite sample with a germanium concentration of about 0.1%, as determined from Raman measurements. All curves clearly display a negative slope.
slope over the entire temperature range examined, indicating n-type conduction. Similarly, a film with \( X_{\text{Ge}} = 54\% \), shown in Fig. 5(c), displays an induced voltage against temperature with a positive slope, indicating p-type conduction. In contrast, a film with an intermediate germanium concentration of \( X_{\text{Ge}} = 18\% \), shown in Fig. 5(b), displays a positive slope near room temperature, and above \( \sim 370 \text{ K} \) the slope of the plot of \( \Delta V \) against \( \Delta T \) has rotated to a negative value. This measurement procedure has been repeated for all of the films synthesized, with \( 0\% < X_{\text{Ge}} < 75\% \).

Fig. 6 shows plots of the Seebeck coefficient against \( 1000/T_{\text{avg}} \) of the nc-Ge/a-Si:H films with germanium crystal fractions ranging from 0\% (pure a-Si:H) up to \( X_{\text{Ge}} = 75\% \). For all of the films for which \( X_{\text{Ge}} < 10\% \), a negative thermopower is observed; fits of these data to \( S = (k_B/e)\left[E_S/k_B T + A\right] \) find a negative activation energy \( E_S \sim -0.3 \) to \(-0.5 \text{ eV} \), consistent with previous measurements of undoped a-Si:H.\(^{20}\) For germanium crystal fractions between approximately 10\% < \( X_{\text{Ge}} < 25\% \), the thermopower exhibits a temperature-dependent transition to positive values at lower temperatures. For \( X_{\text{Ge}} > 25\% \), the thermopower is positive for all temperatures examined, with a smaller, positive slope of \( E_S \sim +0.2 \text{ eV} \).

P-type conduction has been observed in bulk single crystal germanium,\(^{21}\) polycrystalline germanium,\(^{22}\) single-crystal germanium nanowires,\(^{23,24}\) Ge/Si core-shell nanowires,\(^{25-27}\) and Ge nanocrystals embedded within a SiO\(_2\) matrix prepared by co-sputtering.\(^{5}\) In nanoscale materials, p-type transport has been attributed to a hole accumulation layer in the germanium nanocrystals due to acceptor-like surface states.\(^{24,26}\) This is in contrast with hydrogenated microcrystalline germanium, which has been found to have an n-type thermopower with grain diameters as small as 5 nm.\(^{28}\) It is possible that a hole accumulation layer arises due to interfacial effects between the germanium nanocrystal and surrounding silicon atoms or polymers, which are not found in microcrystalline germanium. The transition from n- to p-type conduction seen in these films as \( X_{\text{Ge}} \) is increased is likely due to the formation of extended, percolating chains of germanium nanocrystals. These nc-Ge channels have a lower activation energy than the surrounding a-Si:H, and effectively short out the amorphous silicon matrix at lower temperatures.

Measurements of the temperature dependence of the dark conductivity are consistent with a transition to conduction occurring through interconnected chains of nc-Ge for \( X_{\text{Ge}} > 25\% \). Fig. 7 shows Arrhenius plots of the conductivity \( \sigma \) for samples in Fig. 6. For clarity, only a few of the conductiviy curves have been plotted. For low (\( X_{\text{Ge}} < 10\% \)) and high (\( X_{\text{Ge}} > 25\% \)) crystal fractions, these data are well described by the expression \( \sigma = \sigma_0 \exp[-E_a/k_B T] \), where \( \sigma_0 \) is the pre-exponential factor and \( E_a \) is the conductivity activation energy. For \( X_{\text{Ge}} < 10\% \), \( E_a \sim 0.75 \text{ eV} \), similar to previous measurements of pure, undoped a-Si:H films\(^{29}\) and for \( X_{\text{Ge}} > 25\% \), we find \( E_a \sim 0.4 \text{ eV} \), consistent with other measurements of germanium nanocrystal films.\(^{5}\)

Samples with intermediate crystal fractions show significant curvature on an Arrhenius plot, however. In order to more accurately determine the conductivity temperature dependence, the "reduced activation energy" is calculated, using a procedure developed by Zabrodskii and Shlimak.\(^{30}\) The logarithmic derivative of the conductivity \( w(T) = d\ln\sigma/d\ln T \) is computed, and plotted against temperature on a log-log plot, shown in the inset of Fig. 7.
Thermally activated conduction would yield a slope of $-1/C_0^1$, with different activation energies represented as parallel lines. Zabrodskii plots for the curves in Fig. 7 indicate that conduction is thermally activated with a single activation energy for both the n- and p-type samples, while the transition samples display conduction similar to that seen in the p-type films at low temperature and conduction similar to that seen in the n-type samples at high temperature. The measured conductivity of the $X_{Ge} = 14\%$ film fits very closely to the sum of two activated conduction paths $\sigma = \sigma_1 \exp[-E_1/k_B T] + \sigma_2 \exp[-E_2/k_B T]$, with activation energies of $E_1 = 0.77$ eV and $E_2 = 0.43$ eV, corresponding to a-Si:H and nc-Ge conduction, respectively. Fig. 8 plots the measured conductivity for this film along with the calculated fit and the exponential curves that comprise the fit curve. The inset of Fig. 8 shows the reduced activation energy calculation performed on each of these curves and serves as further evidence of the quality of the fit.

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V. DISCUSSION

The shift from n- to p-type conduction may reflect a transition from transport through the a-Si:H to through the germanium nanocrystals, similar to a percolation transition.
Such a transition is expected to occur at a volume fraction of $\sim 20\%$ for randomly packed hard spheres,\textsuperscript{32–34} though here the observation of this transition is smeared due to variations in nc-Ge concentration within a given sample. Unlike traditional percolative systems, the two phases both conduct relatively equally. The transition region ($10 < X_{\text{Ge}} < 25\%$) in Fig. 6 then arises from the competition between the a-Si:H and nc-Ge conduction channels operating in parallel in the film. In this case, one should be able to fit the observed temperature dependence of the thermopower using a “two-band” expression\textsuperscript{35}

\[
S(T) = \frac{(1 - X_{\text{Ge}})\sigma_{\text{Si}}S_{\text{Si}} + X_{\text{Ge}}\sigma_{\text{Ge}}S_{\text{Ge}}}{\sigma(T)},
\]

where \(\sigma_{\text{Si}}\) and \(\sigma_{\text{Ge}}\) are the conductivities of the pure a-Si:H phase and the pure nc-Ge phase, respectively, \(S_{\text{Si}}\) and \(S_{\text{Ge}}\) are the corresponding thermopower values, and \(S(T)\) is the thermopower as a function of temperature for a given nc-Ge/a-Si:H film in the transition region. The conductivity \(\sigma(T)\) is calculated by modeling the volume fractions of each phase as parallel conducting channels, such that \(\sigma(T) = (1 - X_{\text{Ge}})\sigma_{\text{Si}} + X_{\text{Ge}}\sigma_{\text{Ge}}\), and the resulting thermopower \(S(T)\) is simply weighted by the fractional conductivity.

Fig. 10 shows a plot of the calculated thermopower for films with a variety of germanium crystal fractions using the two-channel parallel conduction model (Eq. (1)) above. The values for \(\sigma_{\text{Si}}\) and \(S_{\text{Si}}\), and \(\sigma_{\text{Ge}}\) and \(S_{\text{Ge}}\), are taken from measurements. For \(10\% < X_{\text{Ge}} < 25\%\), the calculated Seebeck coefficient in Fig. 10 does change from that comparable to pure nc-Ge at low temperatures to that similar to pure a-Si:H at higher temperatures. However, the complete calculated transition is much more gradual than what is actually observed (Fig. 6).

The most notable differences between the calculated (Fig. 10) and measured (Fig. 6) Seebeck coefficients are seen at \(X_{\text{Ge}} < 10\%\) at lower temperatures and \(X_{\text{Ge}} > 25\%\) at higher temperatures. The two-channel parallel conduction model predicts a significant influence of the nanocrystals at \(X_{\text{Ge}} < 10\%\), and a significant contribution from the a-Si:H for \(X_{\text{Ge}} > 25\%\), but neither effect is seen in the data (Fig. 6).

For \(X_{\text{Ge}} < 10\%\), both the measured conductivity and thermopower activation energies are constant and similar to those of pure a-Si:H. In this regime, clusters of nanocrystals will not span the entire sample, and the current will flow predominantly through the a-Si:H. In order for current to pass through any isolated nc-Ge, it must overcome the band offsets between the two materials, as sketched in Fig. 11. This cartoon illustrates the conduction and valence bands at the a-Si:H/nc-Ge heterojunction; the magnitude of contributions to band offsets from Si-Ge alloying, strain fields, and surface charges at the nanocrystal interface is not well known and is not included in this simple sketch. Computer simulations\textsuperscript{36,37} and scanning tunneling spectroscopy measurements\textsuperscript{38} find that the energy gap of 4 nm Ge nanocrystals is approximately 1.4 eV due to quantum confinement effects. The a-Si:H is n-type and the nanocrystals are p-type, therefore current paths that enter and then exit a nanocrystal would effectively see an n-p-n junction. This would significantly increase the resistance of such current paths, resulting in a smaller current through the germanium nanocrystals than predicted by the two-channel parallel conduction model. At some crystal fraction, the clusters would become sufficiently extended for the higher conductivity of the germanium nanocrystals to outweigh the resistance associated with charge transport into the nanocrystal phase, and conduction would begin to flow through the nanocrystals, particularly at lower temperatures, where the resistance of the a-Si:H matrix is high. In addition, even once an extended chain of germanium nanocrystals is formed, the conductivity of this phase may be less than expected from the two-channel parallel conduction model. Many of the nanocrystals may form dead ends or loops which do not contribute to the conductivity of the Ge phase.\textsuperscript{34} Note that in Fig. 8, the magnitude of the p-type conductivity curve needed to fit the data for a transition film is ~30 times smaller than the conductivity of films with \(X_{\text{Ge}} > 25\%\) in Fig. 7.

The disagreement between the observed thermopower and the two-channel conduction model for \(X_{\text{Ge}} > 25\%\) may
be due to the influence of the nano-inclusions on the surrounding a-Si:H matrix. In the two-channel parallel conduction model for samples with $X_{\text{Ge}} < 10\%$, conduction through the a-Si:H should decrease linearly with increasing $X_{\text{Ge}}$, but preliminary results show a much faster decrease. This could reflect increased disorder within the a-Si:H caused by additional compositional morphology from the nanocrystal inclusions. As the a-Si:H conductivity decreases rapidly with increasing $X_{\text{Ge}}$, the component of the two-channel parallel conduction model due to n-type transport in the amorphous matrix should be significantly suppressed at larger germanium concentrations. This is consistent with the measured thermopower and conductivity for all samples with $X_{\text{Ge}} > 25\%$, which show no detectable n-type conduction, unlike the calculated values in Fig. 10. A thorough characterization and detailed analysis of the influence of nc-Ge on the conductivity of a-Si:H will be described separately.

In summary, using a dual-chamber co-deposition system, we have synthesized composite thin films consisting of hydrogenated amorphous silicon in which germanium nanocrystals are homogenously embedded. X-ray diffraction indicates that the nanocrystals are approximately 3.5–4.5 nm in diameter, and RBS and Raman spectroscopy provide quantitative determinations of the germanium crystal fraction in these films. The conductivity and thermopower have been studied as the nc-Ge are systematically varied from 0% to 75%. For films with $X_{\text{Ge}} < 10\%$, conduction is n-type, similar to pure a-Si:H, while for $X_{\text{Ge}} > 25\%$ transport is p-type, as observed in other systems of germanium nanocrystals and nanowires. In the transition region, 10% < $X_{\text{Ge}}$ < 25%, transport is n-type above 400 K and p-type near room temperature. However, the observed temperature dependence of the thermopower for these transition films is much faster than can be accounted for by a simple two-channel parallel conduction model. The differences between the simple model and the measured results can be qualitatively explained as a combination of two effects: Conduction paths through the nanocrystal phase are series of n-p-n junctions for samples with low $X_{\text{Ge}}$, and long-range disorder induced by the nanocrystals decreases the conductivity of the surrounding a-Si:H material. Further studies are underway to elucidate the detailed transport mechanisms in these novel films ordered on the mesoscale.

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31The measured slope in the Zabrodskii plots for these films is slightly less than −1, and will be discussed in detail elsewhere.