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Analysis of Mechanical and Electrical Responses in Molybdenum Silicide Thin Film Laid on PET During Tension Application

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Abstract

The mechanical and electrical behavior of molybdenum silicide (MoSi2) thin films sputtered on Polyethylene Terephthalate (PET) substrates is examined in this work, with a focus on tensile strain. PET substrates were systematically prepared, coated with different thicknesses of MoSi2 (between 100 and 200 nm), and then put through tensile testing to evaluate the mechanical integrity and electrical properties. Scanning electron microscopy (SEM) was utilized to observe the formation and evolution of cracks, particularly noting their density, distribution, and orientation relative to the applied load. The results revealed an increasing pattern in crack density and width heightened strain levels, notably perpendicular to the direction of tension. Thicker films demonstrated heightened resistance to cracking, suggesting a correlation between film thickness and mechanical robustness. Concurrently, electrical resistance measurements indicated a direct relationship between increasing strain and resistance, reflecting the structural disruptions within the film. This research provides critical insights into the fracture mechanics and electrical response of MoSi2 thin films under stress, highlighting the implications for their use in flexible electronics and other applications where endurance and adaptability are paramount.

Keywords: Molybdenum silicide; MoSi2; Tensile test; Stretching; Thin film.

1. Introduction

Energy serves as the cornerstone of technological progress and a critical enabler of modern civilization, with growing demand driving innovation across various sectors [1–5]. In particular, the shift toward sustainable, portable, and efficient energy solutions has underscored the vital role of advanced electronic systems [6–8].

Molybdenum silicide (MoSi2) is a silicone-based composite material that is extensively used in many industrial applications because of its unique characteristics and the flexibility [9]. Hexagonal MoSi2 is a silver-white solid material. It is very rare and it comes from the earth naturally as a molybdenum mineral [10]. MoSi2 can be made synthetically by heating silicon tetrafluoride with molybdenum oxide [11]. MoSi2 is a precious material because of its special features. A few of its significant characteristics include its power and durability, resistance to heat, and ability to transfer heat [12]. MoSi2 has an excellent corrosion resistance and chemical stability that makes it an ideal material in the pharmaceutical and agricultural industries [13].

In application, MoSi2 is a very versatile material that can be applied in many industries. For instance, MoSi2 has often been used in the manufacture of steel and aluminum, as well as in construction materials because of its high strength and durability [14]. The resistance to heat is of great importance in the semiconductor industry due to the fact that an environment of high temperature is objective in so many manufacturing processes [15]. The extraordinary thermal conductivity of MoSi2 finds an essential role in heater manufacturing and cooling mechanisms. Besides this, MoSi2 has high resistance to corrosion and resists chemical attacks, hence is ideal for application in the pharmaceutical and agricultural industries [16]. In the aerospace industry, MoSi2 is useful in making spacecraft and satellite parts because it can bear very high temperatures with high mechanical stability [17]. A significant advancement in high-precision electronics manufacturing is the deposition of $MoSi_2$ thin films onto various substrates. These thin layers can be fabricated using techniques such as chemical vapor deposition (CVD)

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and physical vapor deposition (PVD), enabling tailored properties suited to a wide range of electronic applications. Due to its excellent thermal conductivity and high heat resistance, $MoSi_2$ is widely utilized in the semiconductor industry.

MoSi2 is used in the manufacturing of thermal, mechanical, and optical components in semiconductor applications. Thermal components include capacitors and resistors, which are in demand in semiconductor applications and will facilitate heat transfer efficiency. The components, such as semiconductor carriers and bearings, will play a vital role in preventing material degradation resulting from corrosion and fracture. Optical components, such as lenses and lasers, are important to facilitate efficient light transmission in semiconductor applications. MoSi2 is also highly useful in semiconductor manufacturing tools, such as cutting and electrical discharge machining tools, which ensure high precision in semiconductor fabrication. In the scientific arena, the work goes from deposition of thin films with MoSi2 to preparation of films with improved properties for industrial applications. The following are a few examples of the mechanical properties of MoSi2 and doped MoSi2 [18-21].

The prime interest in MoSi2 and its composites has been in their mechanical behavior and properties arising out of their potential application in high temperature structural environments. [22] gave basic data on the review of the mechanical properties of MoSi2 in its single crystals, polycrystalline forms, and composites, putting emphasis on its deformation and fracture toughness at high temperatures. [23] examined $MoSi_2$-based alloys and composites, emphasizing the effects of strain rate and temperature in the presence of different reinforcing phases, and highlighting how variations in composite additives such as Mo_5Si_3 and CaO influence mechanical strength through size effects. [24] explored the impact of alloying $MoSi_2$ with elements like Re and Al, demonstrating significant changes in properties such as solid solution hardening and the ductility transition temperature. In [25], the relationship between microstructure and mechanical properties in $MoSi_2$–SiCnanolayer composites was investigated, with a particular focus on how heat treatments affect hardness and modulus by promoting crystallization and grain growth. [26] discussed in detail the specific mechanism of MoSi2 formation through mechanical alloying; under certain conditions, this involves a self-propagating high-temperature synthesis. [27] discussed the thermal and mechanical properties of α-MoSi2 in view of its high thermal conductivity and oxidation resistance, therefore being a good candidate for various high temperature applications. [28] gave an ab initio contribution concerning the mechanical stability of MoSi2, discussing the modifications which relevant strains may cause in its electronic structure and stability. [29] investigated MoSi2 coatings for mechanical properties and oxidation resistance, further elucidation of the practical applications and limits of materials based on MoSi2. In [30] was investigated the mechanical behavior of the ZrB2-MoSi2 ceramics, evidencing how MoSi2 content and ZrB2 grain size interactively influence the mechanical properties of microhardness and fracture toughness. Together, these works formed a complex story about the mechanical behavior and properties of MoSi2; it underscored the versatility of this material in high-temperature and high-stress conditions.

This review described the development of knowledge about MoSi2 and underlined the synergistic effect of different modifications, treatments, and composite formations on mechanical properties. On the other hand, synthesis approaches and property enhancements in MoSi2 are also addressed. Similarly, [31] explored the effect of MoSi2 mechanical alloying on its microstructure and mechanical properties in spark plasma sintering. Their work underlined the importance of mechanical alloying time for the formation of different phases and how it could affect the thermodynamics of the reactions. [32] continued the study of the phase stability and mechanical properties of MoSi2 alloyed with Al, Mg, and Ge by means of first-principles methods.

Their studies improved the understanding of how alloying elements modulate the phase transition and, hence, ductility and hardness. Correspondingly, the review by [33] on the phase transformation and mechanical properties of sputter-deposited MoSi2 thin films emphasized amorphous-to-crystalline structure transformations and their respective mechanical properties. [34] discussed the mechanical properties of MoSi2 coatings deposited onto Mo substrates by hot dipping processes. The emphasis was on how the temperature and dipping time influenced the mechanical performance. [35] presented laser-processed MoSi2 coatings, describing process parameters that relate to the microstructure and mechanical properties of the coatings. They presented the difficulties in obtaining coatings free of cracks and solutions. In a study on high-pressure sintering of bulk MoSi2, [36] investigated the effect of temperature and high pressure on mechanical characteristics, microstructural development, and sintering behavior. Their findings revealed how high-pressure sintering can attain outstanding mechanical properties such as hardness and fracture toughness. In [37], the authors investigated the mechanical behavior of MoSi2-reinforced Si3N4 matrix composites, focusing on the effect of the content and size of the MoSi2 phase as well as of the MgO densification aid on the mechanical performance. Nanocrystalline MoSi2-SiC composites have been synthesized in [38]. A detailed dependence of hardness and fracture toughness is shown, especially on the average crystallite size and on the absence of particular phases. [39] considered enhancement in mechanical properties in MoSi2 by additions of Sc2O3 and Y2O3 and provided some insight into how such low-level additives can realize great improvement in strength and fracture toughness. In addition, the study by [40] focused on the Re and Al alloying effects on the mechanical properties and high-temperature oxidation of MoSi2, giving some clues on mutual interaction of these elements in regard to strength and ductility improvement. [41] The consolidation and mechanical properties of MoSi2-based materials were investigated, and HIP was applied to manufacture compacts with minimum porosity and optimum microstructures. The paper emphasized the role of consolidation parameters on the mechanical behavior. [42] Mechanical and microstructural characteristics of metal/ceramic microlaminates of Nb/MoSi2 systems have been presented, which demonstrate that layer thickness has an impact on hardness and modulus. [43] discussed the influence of MoSi2 content on dielectric and mechanical properties of MoSi2/Al2O3 composite coatings and highlighted the trade-offs existing between the mechanical properties and the dielectric performance. [44] fabricated MoSi2-matrix composites that were reinforced with SiC and ZrO2 particles and discussed such reinforcements' synergy in mechanical properties.

Finally, [45] examined the effects of carbon addition on the mechanical properties of the MoSi2-TiC composites. It illustrated that carbon can significantly improve the bending strength and fracture toughness by eliminating the glassy SiO2 phase. Through this compilation of studies, a complex picture is built up regarding the various strategies and techniques used in manipulating and enhancing the mechanical properties of MoSi2 and its composites. Starting from alloying and sintering through coating to reinforcement, each method gives insights into optimization of MoSi2 for applications requiring high performance. Coupled together, the evolution of synthesis methodologies with basic understanding of material behavior is paving the way to develop MoSi2 materials with tailored properties for specific needs and applications. The work here describes the mechanical and electrical behavior while focusing on the tensile strain of MoSi2 thin films sputtered onto PET substrates. Coat PET substrates with different thicknesses of MoSi2 in the range between 100 and 200 nm and then subject the film to tensile testing to evaluate the mechanical integrity and electrical attributes which resulted. The present research studiously explores how stretching affects the layers deposited on the substrate, especially MoSi2, in its electrical and mechanical properties.

2. Research Methodology

First, test specimens of PET substrates were cut into pieces, each 127 micrometers thick, and sectioned into uniform pieces using a sharp precision knife. These pieces were thoroughly cleaned and dried in preparation for the subsequent processes. The pieces were then positioned in a sputtering chamber fitted with a target of MoSi2. RF magnetron sputtering was performed with the following parameters: RF power of 250 W, argon gas flow at 29 sccm, keeping the vacuum pressure less than or equal to 2.5 x 10^-3 Pa, and the substrate rotated at 20 rpm. Under these conditions, a 100 nm-thick MoSi2 coating was deposited in one hour and a 200 nm coating in two hours.

Immediately after coating, each sample was mounted with care on an Instron tensile testing machine following a standardized procedure. The samples were fixed within the jaws of the device, set 80 mm apart from one another. Electrical contacts in the form of copper tapes were fixed 10 mm away from each end of the jaws on the samples and attached with tweezers that were connected to these tapes. These tongs were attached to an ohmmeter that was programmed to measure electrical resistance in real time and to store the readings in a computer directly. The experimental setup is shown in Figure 1.

The tensile tests were done by steadily raising the upper jaw of the Instron machine at a constant rate of 1 mm/s until sample failure. Each test was run four times per thickness to ensure the reliability of the results. Some of the specimens were stretched up to different elongations - namely, 7.5%, 10%, and 12.5% of the original length - both with and without connection to the ohmmeter, and subsequently tested with a Scanning Electron Microscope (SEM) in order to closely look at and record changes onto the sample surfaces.

3. Results and Discussion

A100 nm MoSi2 thin film was deposited on 127 µm-thick PET substrate. The structure was then stretched at different strain rates. The thin film surface was observed at strains of 7.5%, 10% and 12.5%, as shown in Figures 2, 3, and 4, respectively. Cracks perpendicularly to the load direction were observed.

The stretched MoSi2 thin film was strained by 7.5%. An amplified, close-up view of the SEM image revealed the formation of cracks which now appeared, from the perspective of the direction of the applied load and crack growth as shown in Figure 2. These cracks are distributed uniformly, at an approximate 90° to the direction of the applied load. The image also shows topographic variations on the specimen, such as ridges and valleys, which were shown by contrast variations on the film surface. The film microstructure is responsible for the mechanical characteristics of the structure. In addition, blow holes caused by sputtering act as initiation sites of the cracks that run parallel to each other, oriented in the direction of the applied load. Figure 2 explains in detail the realization of such cracks within the layer of thin film.

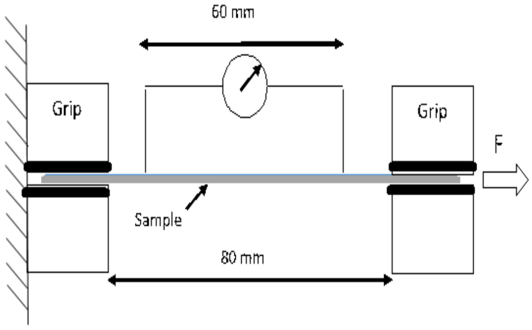

Figure 1. Schematic diagram of the specimen and grips.

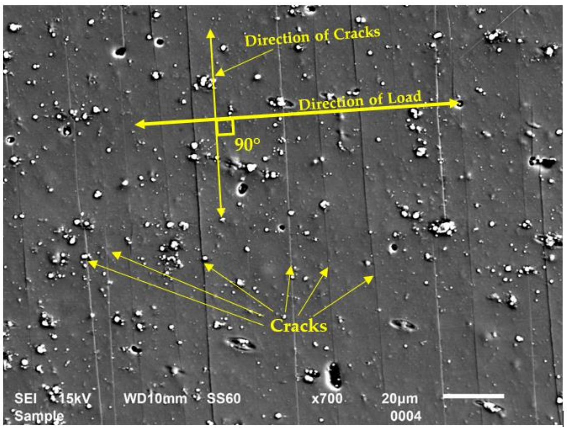

Figure 2. Initial Crack Formation in 100 nm MoSi2 Film at 7.5% Tensile Strain

Figure 3 depicts the result for stretched MoSi2 thin film with 10% strain. It is observed from this figure that from the surface imperfections, fine cracks emanate and grow perpendicular to the load direction. The crack nucleation process appears to be accelerated by the surface imperfection according to the many initiation sites of surface cracks on the surface. While Figure 2 presented a distribution of cracks that was consistent with much smaller cracks, Figure 3 shows more pronounced and dense cracks, reflecting increased damage. Therefore, it is expected that the value of strain is higher, resulting in more severe deformation leading to a loss of structural integrity in the thin film. The comparison of data in those figures gives an indication of the dependence of strain with regard to damage in film, which would be essential in setting the mechanical threshold necessary in preventing failures in application design involving similar materials under uniaxial tensile stress.

Contrasting that, Figure 4 depicts the MoSi2 thin film subjected to stretching up to 12.5%. In this figure, the crack pattern was more severe as compared to Figures 2 and 3. The cracks were apparently wider, and by this, it could be seen that the film under tensile stress had faced a higher magnitude than previously discussed, hence leading to more critical damage. A number of these cracks seemed deeper and extended through the entire thin film, hence suggesting even lesser structural integrity.

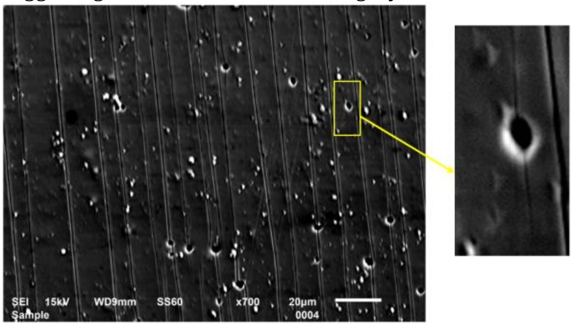

Figure 3. Progressive Crack Development in 100 nm MoSi2 Film at 10% Tensile Strain

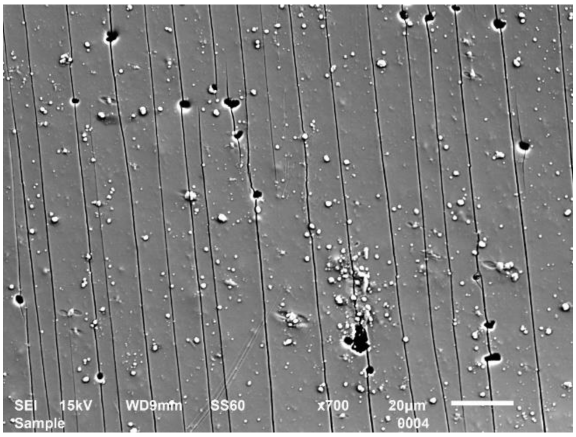

Figure 4. Advanced Crack Propagation in 100 nm MoSi2 Film at 12.5% Tensile Strain

It can be observed from Fig.4 that, upon stretching of MoSi2 thin film up to 12.5%, the role of imperfections, such as holes, became stronger. In general, these imperfections in materials science often serve as a stress concentrator where the local stress at a point around that imperfection is higher than that of its surroundings, thus making them natural initiation points for failure. These stress concentrators can initiate cracks when a material is under tension and would explain why the holes are associated with crack initiation in the image provided. Some of the smaller cracks may not propagate due to a reliving of some of the stress concentration around the holes by the larger cracks themselves. This phenomenon is related to the principle of crack shielding whereby the presence of a major crack can shield other potential crack sites from the full stress field, hence reducing their likelihood of growing.

The observation that some cracks deviate and stop could be a result of several factors. It could be due to regions in the material with different microstructural properties resisting crack propagation. These can be due to inhomogeneities within the material composition, inclusions, or from areas where the bonding to the substrate is more extensive. Another cause may be the interaction of crack paths: when two cracks partially overlap, the interaction may distort the stress field so that further propagation of one or both cracks is prevented. The crack that appears to be deflected and arrested demonstrates the complexity of fracture mechanics in heterogeneous materials. Their deviation may depend either on finding different mechanical properties or on interaction with other cracks or boundaries within the material. Arresting of a crack implies that the local intensity of stress has fallen below the threshold value required for further crack growth, which could be a consequence of dissipations of stress, crack path deflection, or presence of toughened regions.

The behavior of cracks in the thin film during stretching may give valuable information about the fracture toughness of the material and on the mechanism of crack propagation and arrest, critical in predicting performance and reliability in practical applications. Figure 5 is a low-magnification image of the 100 nm MoSi2 thin film on 127 µm PET substrate after a 12.5% stretch. This wider view allows observation of both cracks and delamination/peeling of the film from the substrate. Such phenomena already mean extensive damage with both mechanical integrity and functional performance implications.

Delamination, in this context, refers to the separation of the layers in laminated composites. It could either be in relation to poor adhesion between the film and substrate or forces caused by stretching, which is higher than the strength of the adhesive. The differences in extensibility between PET and MoSi2 will result in shear stresses in the film as it is stretched, possibly causing delamination. Peeling, which refers to regions where the film has lifted off, is the effect of the film not only delaminating but also curling or flaking away from the substrate. It might be because of a critical breakdown when the material can no longer support its structural cohesiveness under the applied strain either in the interface or within the thin film itself. These types of damages are significant on a scientific basis because they indicate a failure mode that is reliant both on the interfacial strength between the film and its substrate aside from the material's inherent tensile strength. It would also infer that in applications dealing with MoSi2 thin films, the adhesive properties and the choice of substrate would be highly relevant, especially when the material will be subjected to mechanical stresses.

This extent of delamination and peeling highlights the mechanical failure mechanisms of failure in thin film composites. It represents the dimensions of material characterization and stress testing, accounting for not only material properties but also the interfacial dynamics between different layers in composite structure properties.

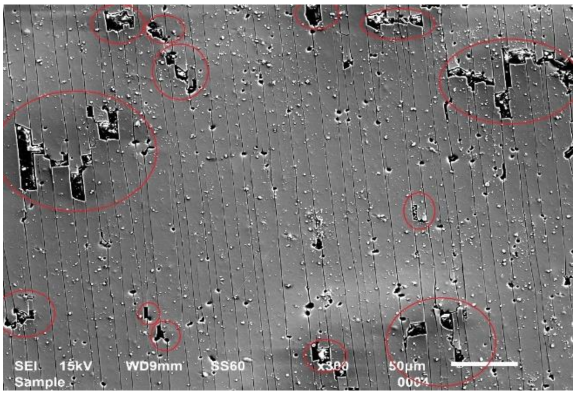

Figure 5. Overview of Delamination and Peeling in 100 nm MoSi2 Film at 12.5% Strain

In another experiment, a 200 nm MoSi2 thin film was deposited on a 127 µm- thick PET substrate and stretched to 7.5%, 10%, and 12.5% of its initial length, respectively.SEM images of the deposited films were obtained after stretching to 7.5%, 10%, and 12.5%, as shown in Figures 6, 7, and 8, respectively. It can be noticed that the surface of the sample which was stretched to 7.5% had less density of cracks compared to the 100 nm samples. This could be due to the fact that increased thickness leads to greater resistance to crack initiation and propagation. Compared to those in Figure 7, the density of cracks is increased at the samples stretched to 10%, but they are still fewer and less severe than those observed in the 100 nm film at the same strain level. The thicker film may possess a higher threshold for crack formation due to the larger volume of material able to absorb and distribute the applied stress.

Figure 8 shows a stretching level of a12.5%. A marked contrast with the 100 nm film under the same conditions was observed. Not all the holes were seen to lead to crack formation in the case of the 200 nm film, an indication of increased resistance to crack initiation around imperfections. This was attributed to the fact that increased thickness provides a buffer against the stress concentration effects that the holes typically introduce. It is also possible that the thicker film has a larger 'bridging' effect-the material between holes can keep its integrity even when some neighbouring regions have cracked. Relatively, the 200 nm film is mechanically more robust than that of 100 nm thickness. The increased thickness allows a higher load carrying capacity and minimizes the possibility of crack nucleation and growth. This comparison exemplifies how film thickness dependence of mechanical properties of thin films is such that a thicker film can enhance fracture resistance at the possible expense of flexibility/weight issues in functional applications.

That not all holes in the 200 nm thick film generate cracks at a 12.5% stretch infers that interplay between material thickness, inherent toughness, and the presence of defects can be complex. Material around such defects redistributes stress in thicker films more effectively to avoid crack propagation and provides a route by which the structural integrity of the film can be maintained at higher strains. This represents a good insight into the design of the thin film material; it could suggest that, at least to a point, increased thickness is potentially one strategy to improve durability without compromising any other needed properties, such as flexibility or transparency.

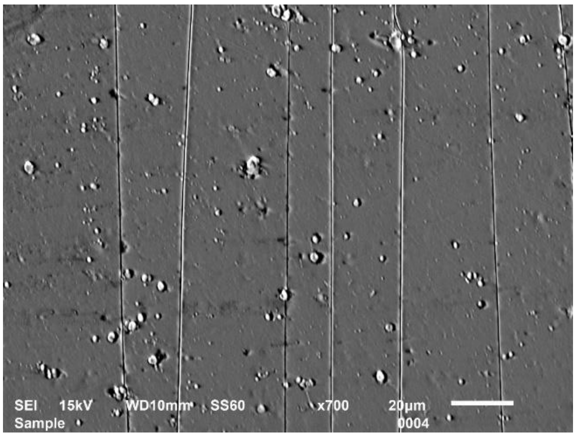

Figure 6. Crack Onset in 200 nm MoSi2 Film at 7.5% Tensile Strain

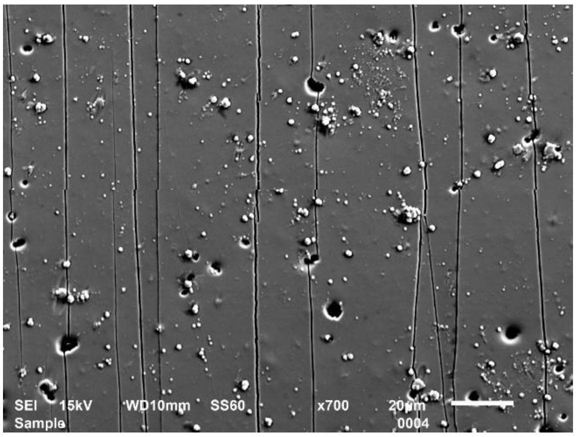

Figure 7. Increased Crack Density in 200 nm MoSi2 Film at 10% Tensile Strain

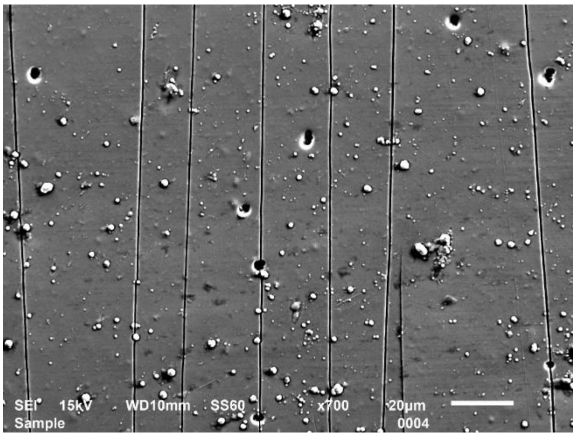

Figure 8. Crack Patterns and Hole Interaction in 200 nm MoSi2 Film at 12.5% Tensile Strain

Figure 9 shows the relationship of strain versus. PCER for a 100 nm MoSi2 thin film. There are three curves shown on the graph representing an average, maximum and minimum PCER out of four tested samples up to resistance increase by a factor of 100.The Figure reveals that while the strain increases, the PCER also rises proportionally. Initially, the resistance increases gradually with the revealed fact that due to minor deformations, the electrical properties of the film remain intact. When the strain proceeds further, the rate of change in resistance increases, and steepening curve in this case would mean that probably microcracks have compromised the structure and disrupt the conducting paths of electricity. The gap between the maximum and minimum PCER curves is due to the variability within the samples that arises, among other things, from variations in crack propagation or from defects.

This increase in PCER corresponds to observations from the SEM images where initial cracks and imperfections were observed as the film was stretched. These changes in structure impact the conduction capability of the film, wherein crack formation results in steep rises in resistance. The SEM images showing more severe cracking at higher strains correspond to the sharp increase in PCER at corresponding strain levels.

Figure 10 also shows the relationship between strain and PCER for 100 nm MoSi2 thin film. The average, minimum and maximum PCER curves were plotted. Comparing Figs. 9 and 10, it is to be noticed that the overall variation of PCER stands on a growing trend with the strain, although some variation in specific value and spread between maximum and minimum PCER may be present. The variability reflected in these graphs explains the observations made on SEM, wherein the presence and propagation of cracks were not consistent among samples. These electrical measurements further quantify physical damage, as depicted in the SEM images, and give an insight into material behavior while under strain. The same resistance measurements reflect the damage from a functional perspective, contrasting with structural insights obtained in the SEM analysis.

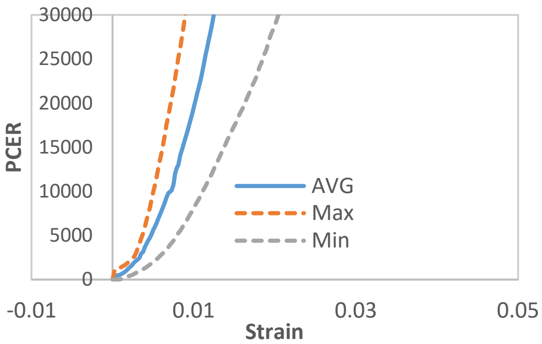

Figure 9. The Relationship Between Strain and PCER for 100 nm Mo Thin Films

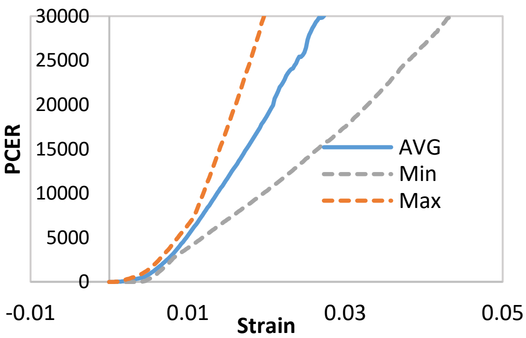

Figure 10. The Relationship Between Strain and PCER for 200 nm Mo Thin Films

4. Conclusions

In the end, this detailed mechanical and electrical study of MoSi2 thin films under tension provided valuable insights into several key aspects of its behaviour and characteristics. The mechanical tests conducted showed the crack formation pattern in the films to a large extent as the number, width, and severity of cracks increased with the increasing degrees of elongation subjected to the films. It was pointed out that these films had a proneness for crack development perpendicular to the applied load, which characterizes their material response to the applied stresses. Significantly, it was seen that the greater the thickness of the MoSi2 films, the higher the resistance to cracking; this indicates that one of the important parameters in enhancing the mechanical robustness may be the film thickness itself. The electrical resistance measurements provided a practical perspective on the issue, revealing that resistance increased with applied strain, particularly at higher strain levels. This rise in resistance closely aligned with the observed physical evidence of crack formation and propagation, highlighting the significant impact of mechanical deformation on the electrical conductivity of $MoSi_2$ thin films.

These results highlighted the great interplay between the material properties, structural integrity, and functional performance in flexible and durable applications. Insights into the tensile-stress-induced behavior of MoSi2 obtained in this work form a background for continued research and development of durable thin films on flexible substrates for electronics applications. Further refinement in the deposition process, improvement in film-substrate adhesion, and understanding the role of microstructural features are other aspects of possible research that might achieve better performance and a wider application range in the use of these materials.

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