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Investigation of “fur-like” residues post dry etching of polyimide using aluminum hard etch mask

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ABSTRACT

The authors found that oxygen plasma etching of polyimide (PI) with aluminum (Al) as a hard-etch mask results in lightly textured arbitrary shaped “fur-like” residues. Upon investigation, the presence of Al was detected in these residues. Ruling out several causes of metal contamination that were already reported in literature, a new theory for the presence of the metal containing residues is described. Furthermore, different methods for the residue free etching of PI using an Al hard-etch by using different metal deposition and patterning methods are explored. A fur-free procedure for the etching of PI using a one step-reactive ion etch of the metal hard-etch mask is presented.

1. Introduction

Polyimides (PI) are thermosetting ring chain polymers comprising of repeating chains of imide monomers. Polyimides are extensively used in Micro-Electro-Mechanical Systems (MEMS) devices because of their outstanding properties such as excellent chemical resistance [1], high thermal stability [2], high mechanical strength [3,4] and good dielectric properties [5,6]. Polyimides are used as sacrificial layers, structural layers, isolation layers and as substrate material in flexible/stretchable electronic circuits [7]. A good example of a technology that makes use of many of the aforementioned properties of PI is the Flex-to-Rigid technology (F2R) [8]. F2R was developed to assemble complex electronic systems, such as for ultra-sound imaging, on the tip of smart-catheters. It enables the fabrication of arbitrary shaped Si islands containing sensors and electronics that are fabricated by through-wafer DRIE etching and that after etching remain suspended in the silicon wafer by tiny PI tabs (Fig. 1). The rigid silicon sensor islands are interconnected using stretchable metal interconnects embedded in the PI. Here the PI acts as a substrate as well as an isolation layer for the interconnects. Dry etching of this PI layer renders the embedded interconnect layer free. These free standing interconnects [9] can bend out of plane when stretched, increasing the stretchability of the device in Fig. 1.

Polyimides can be of two types: photosensitive and non-photosensitive. In this paper, only non-photosensitive polyimides will be addressed. Patterning of the PI can be achieved by using either a resist mask or a hard-etch mask. In general, the selection of a mask is based on firstly, the selectivity of the etch process towards the mask, and secondly, its ease of integration as a masking material in the flowchart. Polyimide is usually dry-etched in gas mixtures primarily containing oxygen. This makes the selectivity of the etch towards resist very poor so that consecutively the mask erodes rapidly during the plasma etching. The selectivity of the PI etch with a photoresist mask is 1:1, implying that the PI and resist layers are etched at an equal rate. This requires the resist mask to be at least as thick as the underlying layer of PI. Thick resists, however, have a limitation for high resolution lithography, for both positive and negative tone resists. An alternative that has been adopted in the microfabrication industry, is the use of a hard-etch mask. In this approach, a metal layer like Al, Ti, Mo etc. is used as a mask for the patterning of the underlying PI layer, where the mask is unaffected by the plasma chemistry of the PI etch. However, according to literature [10], the adhesion of a metal layer to an untreated surface of polyimide is poor due to its low specific surface energy. For a proper adhesion of the metal mask to the PI, a short Ar^+ sputter-etch of the polymer surface is recommended [10]. The metal layer is then patterned using a resist mask in the desired shape for the etching of PI. Prior to the etching of PI, the photoresist is stripped in acetone.

The plasma chemistry used for the etching of PI consists of oxygen radicals which break the unsaturated groups within the chemical structure of the polyimide. After etching of the polyimide using an Al metal mask, residues on the etched areas were observed. These residues were arbitrary in shape, and appeared light in texture, and for ease of

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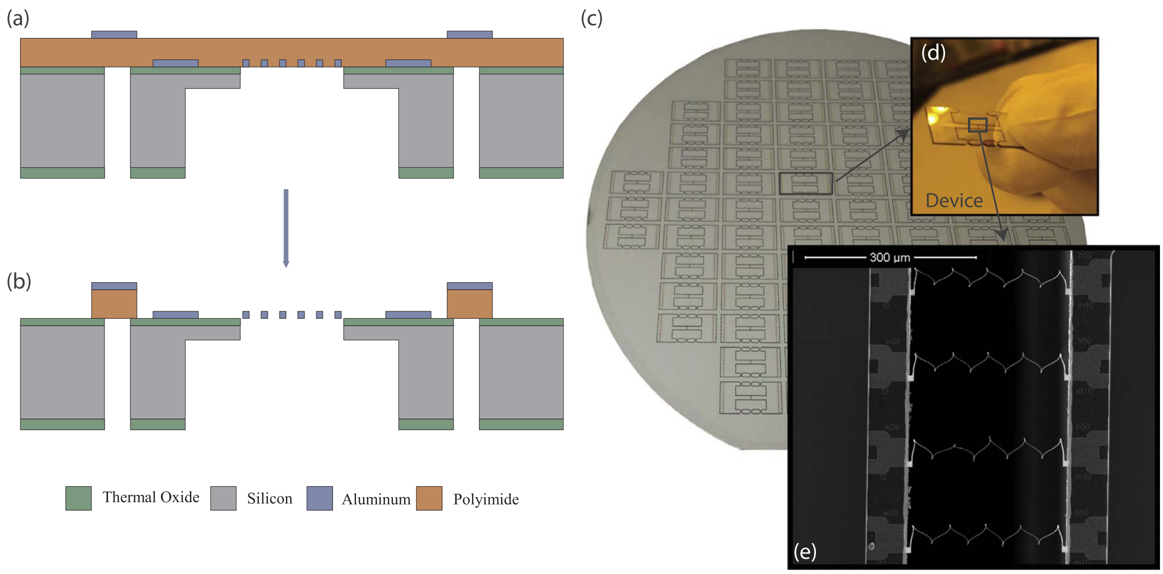

Fig. 1. On the left, flowchart depicting the cross section of a wafer for the fabrication of free standing interconnect structures where (a) the metal interconnects are embedded in PI. After the reactive ion etch (RIE) etch of PI, (b) the interconnects are rendered free standing while the PI tabs remain. On the right, (c) a 6-in. test device wafer with the sacrificial PI tabs holding the devices together after the RIE etch. These tabs can be easily cut using laser to (d) isolate the device from the wafer. Upon stretching of the device, as seen in the SEM (e) the free standing interconnects bend out of plane to enhance the stretchability. The device is elaborately explained in the literature from Shafqat. S et. al. [9].

understanding will be referred to as fur-like residues in this paper. Similar residues have been reported in the literature [11,12], wherein the silicon content in the self priming and silicone modified polyimides played a role. In this paper, however, we do not use either of the polyimides, thus eliminating the cause of the aforementioned occurrence of the residues after reactive ion etching. The presence of these residues is undesirable as it leads to process instability and may interfere with the subsequent microfabrication steps. An interesting outlook towards these residues could be to harness them for the formation of nanowires, black silicon or in the Bosch process by controlling the residue size and density. However, it will be not be discussed in this paper as it is beyond the scope of this study.

The goal of this work is to investigate the origin/nature of these "fur-like" residues and provide solutions compatible with the microfabrication of PI-based electronics. Different hypothesis explaining the origin of the fur-like residues were tested by variations in process conditions and material analysis techniques (SEM, EDX, AFM and Raman). The experimental section describes the general processing flow employed to realize the PI-based devices/structures and motivation for the several short loop experiments and hypothesis. The subsequent sections discuss results for each hypothesis. Finally, devices are demonstrated that were fabricated through improved processing conditions, which eliminated the fur-problem, while maintaining compatibility with advanced MEMS-processing flows.

2. Experiment

The presence of the residues was first observed during the fabrication of a device wafer with test structures to characterize free-standing interconnects [9] (Fig. 1). The wafer has a pre-defined and patterned 5 µm PECVD SiO_2 hard etch mask (Novellus PECVD concept one) on the backside. This back oxide mask is used to completely etch through 400 µm of Si (30 min in $SF_6 + C_4F_8$ DRIE plasma, STS ICP tool) on certain predefined locations such as to render the top metal structures free from the underlying substrate (Fig. 1-a). These top metal structures are embedded in a 5.2 µm thick PI (Dupont 2611) layer which is spin coated at 3000 rpm for 45 s and cured at 275 °C for 3 h in a nitrogen oven (KOYO Thermo Systems Co. Ltd., Japan). This polyimide layer acts as both sacrificial layer to embed and protect the final free standing interconnect structures, as well as for sacrificial tabs (Fig. 1-b). These tabs keep the devices attached to the wafer and are released by laser or scalpel cutting (Fig. 1-c and d). To obtain a good adhesion between polyimide and Al hard etch mask, the surface of polyimide is sputter etched in an Ar^+ ion plasma (50 sccm Ar gas, 300 W, 100 s in Veeco 2 Nexus, UHV system), where the charged Ar^+ ions accelerate towards the PI surface creating micro roughness on the surface as well as chemically modifying the PI structure [13]. This is done to ensure a good chemical as well as mechanical bonding of metals like Al, Cu, Ni etc. to PI, which can be challenging due to its low surface energy [14]. Next, a 200 nm layer of Aluminum is sputter coated (Veeco 2 Nexus, UHV system with 99.99999% Al target purity and 2 nm/sec deposition rate) on top of the PI layer. This Al hard etch mask is patterned using a photoresist (PR) mask (HPR504 positive resist, OCG Microelectronic Materials n.v) with a thickness of 1.7 µm. The resist mask is spin coated on an automated spin coater (EVG 150, Austria), exposed with an energy of 160 mJ (ASML PAS5500, The Netherlands) and developed in a TMAOH based developer. The Al hard etch mask is wet etched in a commercially available wet etchant PES 77-19-04 (heated etch at 30 °C) consisting of phosphoric acid H_3PO_4, nitric acid HNO_3 and acetic acid CH_3COOH with an etch rate of 95 nm/min. Subsequently, the resist mask is removed in acetone (CT60 spin coater, Gyrset). This hard etch mask is used to etch the PI in an O_2 plasma (6 min RIE in STS ICP tool). After the PI etch, "fur-like" residues were observed under the scanning electron microscope (SEM) around the free standing as well as not-free standing structures (Fig. 2-a and 2-b). This sample will be denoted as the reference sample (*R*) in all the preceding experiments.

For the investigation into the origin of these residues four different experiments are prepared. Each experiment is tested for a different hypothesis. The sample substrates in all the experiments were 6- inch Si wafers coated with 1 µm of plasma enhanced chemical vapor deposited (PECVD) SiO_2 (Novellus PECVD concept one). In all the experiments, the samples have been spin coated with 5.2 µm PI and the PI is etched with similar conditions as the reference sample. The process variations for different experiments along with the hypothesis and their consecutive results are listed in Table 1. The choice for these process variations and their stated hypothesis is elaborated further in the discussion section. Each experiment is repeated three times. The results were the same for each.

In the first experiment, the formation of residues due to metal redeposition is tested. The Al hard etch mask in the sample A is processed