MATERIALS RESEARCH CORPORATION
Advanced Materials Division
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THE ROLE OF TUNGSTEN-TITANIUM TARGET DENSITY ON PARTICULATE GENERATION

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Summary-
Current powder metallurgy manufacturing techniques are capable of producing tungsten-titanium (W-Ti) sputtering targets which generate low particulate levels. Data from MRC and other target manufacturers indicate that particulate generation can be reduced by maximizing the density of the target. This parameter appears to be independent of manufacturing technique.

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ABSTRACT

Current powder metallurgy manufacturing techniques are capable of producing tungsten-titanium (W-Ti) sputtering targets which generate low particulate levels. Data from previous publications by MRC and other target manufacturers indicate that particulate generation can be reduced to 0.2 particles per square centimeter or better by maximizing the density of the target. This parameter appears to be independent of manufacturing technique.

INTRODUCTION

Particulate generation during the use of W-Ti sputtering targets is the subject of a number of recent studies by target manufacturers. These targets are made by a variety of powder consolidation methods including: inert gas hot pressing, vacuum hot pressing, hot isostatic pressing, and cold pressing/sintering. Advocates of the various techniques have each emphasized the value of their particular equipment or methods in minimizing particulates. Several recent studies are based on broad assumptions and on data that is not related to the actual semiconductor manufacturing experience.

Waterman et al.[1] and Dunlop et al.[2] compare hot pressing, vacuum hot pressing and hot isostatic pressing, concluding that vacuum hot pressing is the preferred technique. Wickersham et al.[3] suggested that hot isostatic pressing is superior to the cold press and sinter technique. These groups propose mechanisms to explain the improved sputtering performance claim. In each case, however, the density of the target is not given sufficient attention as a critical parameter for particulate generation.

The purpose of this paper is to review previous work which relates target characteristics to particle generation, and the effects of microstructure and density on particulate generation from W-Ti sputtering targets.

EXPERIMENTAL PROCEDURES

Particle measurements were conducted at operating semiconductor production facilities using a Tencor Surfscan 5000. The technique involves laser beam scattering from the coated surface to detect and count particles.

A scanning electron microscope was used to examine the microstructure of W-Ti sputtering targets. The technique provides atomic number contrast to distinguish between phases that are rich in either tungsten or titanium

X-ray diffraction, employing a computer-controlled Rigako DMAX system, was used to detect phase differences. Samples were scanned from 30’ to 110’ of 2θ in 0.2° steps, with a 0.4 second count time.

RESULTS and DISCUSSION

The data of Wickersham et al. are plotted in Figure 1, along with the window of data collected from MRC ECLIPSE™[4] W-Ti targets. Wickersham used hot isostatic pressing to consolidate the part, using both temperature and pressure as variables. These variables can influence the density of the target. Figure 2 compares the target densities with particle data as reported by Wickersham. These data show a direct correlation of particle count with target density. The MRC data included in Figure 2 indicate that consolidation temperature alone is not the critical variable. The results agree with previous MRC data[5] indicating that the use of higher density targets can result in lower particulate levels. Such a comparison was not developed or examined by Wickersham, although target density is the critical parameter.

Both Dunlop and Wickersham attempt to correlate the existence of a "β(Ti,W)" with particulate generation. Dunlop performed microstructural analysis with X-ray diffraction and energy dispersed X-ray analysis. These techniques can confirm the phases and structure present. However, the findings are poorly interpreted. Wickersham assessed the amount of "β(Ti,W)" with the optical microscope. This technique is only acceptable as a supporting technique when used with others to distinguish between phases. Thus, because a clear interpretation of the microstructure of W-Ti sputtering targets has not been developed, a definition is necessary of what is meant by "β(Ti,W)" and what, if any, role it plays in the generation of particulate from sputtering targets.

DEFINING "β(Ti,W)"

"β(Ti,W)" is an area in the microstructure consisting of finely dispersed tungsten within titanium. Thus, instead of one "phase," it actually contains two phases. This microstructure is the result of a solid-state precipitation, a eutectoid reaction. This can be explained with the use of the W-Ti phase diagram[6], Figure 3.

Solid tungsten exhibits a body centered cubic (bcc) structure from room temperature to its melting point. Pure titanium has an hexagonal, closed-packed (hcp) structure at room temperature, and transforms to bcc at 885°C. The phase diagram indicates that, for temperatures above 740°C, a 10% (weight) titanium alloy will consist of a single a-phase solid solution. The inherent assumption in the use of phase diagrams is that a state of equilibrium is attained. Achieving equilibrium for a W-Ti system requires sufficient time at elevated temperature for diffusion to homogenize the distinct regions of titanium and tungsten that exist in the original powder. To varying degrees, this diffusion occurs during consolidation to establish local regions of near equilibrium compositions.

In commercially available sputtering targets, equilibrium is not attained since tungsten and titanium are not uniform throughout the structure. Diffusion of tungsten into regions of titanium tends to produce areas represented by the titanium-rich region of the phase diagram. As these titanium-rich areas cool, the solubility of tungsten in titanium drops, precipitating tungsten. This fine distribution of tungsten in the surrounding titanium is called "β(Ti,W)".

Figure 4 shows the microstructure for an MRC target and a vacuum hot pressed target. These secondary electron images show contrast by atomic number differences between titanium (atomic number 22) and tungsten (atomic number 74). Figure 4a shows the distinct phases of titanium (dark areas), and tungsten (light areas).

Further independent confirmation of the phases present in W-Ti targets is shown in Figure 5. X-ray diffraction patterns from samples of various commercial W-Ti targets were collected. The results show the existence of only one bcc phase, while the phase diagram predicts two, and metallography shows two distinct phases. This apparent contradiction can be explained by the similarity of the lattice parameter, a,, which is the specific distance between neighboring atoms in the structure for both bcc titanium and tungsten.

	25%	900°C
Ti	$a_o = 2.95Å$ $c = 4.686Å$	$a_o = 3.30Å$
W	3.165Å	

Lattice Parameters

The fact that a-titanium (hcp) is not detected by X-ray diffraction at room temperature suggests that tungsten acts like other known p-phase stabilizers. Elements including molybdenum, tantalum, vanadium and niobium act to preserve the bcc phase of titanium at room temperature. From the results in Figures 4 and 5, the type of microstructure that Wickersham claims is "β(Ti,W)" is likely to consist of stabilized β-titanium with tungsten precipitates.

The microstructure in Figure 4b is from vacuum hot pressed material and shows no reaction products in the titanium rich areas. The quantity of tungsten dissolved and diffused into titanium during consolidation was not sufficient to exceed the solubility of tungsten in titanium. However, from the X-ray data shown in Figure 5, the quantity of dissolved tungsten is sufficient to stabilize the bcc form of titanium at room temperature and appears to be isomorphic with tungsten.

PARTICLE PERFORMANCE

Combinations of the amount of "β(Ti,W)" and target density were the basis of Wickersham's argument in reference 3. The investigators evaluated two combinations for particle performance: 1) low density with high "β(Ti,W)," and 2) high density with low "β(Ti,W)." They neglected to assess two other possibilities: 3) low "β(Ti,W)" content with low target density, and 4) high "β(Ti,W)" content with high density.

The MRC Eclipse target exhibits combination #4, above. The fact that this target generates low particle levels in the range of less than 0.2 p/cm^2 indicates that low "β(Ti,W)" is not required for acceptable particle performance. Although no data are reported or available, combination #3 is expected to generate high particle counts.

The authors of reference 3 contend that particle generation from the sputtering target is significant only during the initial stage of target life, while particle levels during the remainder of target life are dominated by "flaking" in the sputtering system. The data from reference 3 represents an initial period of target life (about 200 kW-hr). But these data were obtained from targets sputtered in a laboratory sputtering system.

For modern sputtering systems, the "incubation period" for flaking is the time during which particle generation is predominantly related to the target. During this period, the generation of particles is lowest. The techniques for extending the incubation for flaking are: specially designed and treated shielding; slow roughing and venting flows to and from vacuum; filtered gases; and special handling of wafers from the cassette to loadlock. Flaking can also be reduced with proper system maintenance. The combined particulate performance over the life of the target is influenced by the design of the system, the quality and frequency of maintenance, and the target density.

Figure 6 shows particle data for an MRC **ECLIPSE-Star™** [4] sputtering system with MRC Eclipse W-Ti targets. The data were collected from bare wafers cycled through the system with process gas flowing, but without sputtering (termed “walk with gas”). The chart shows the mean particle level on a 150 mm wafer to be 0.08 p/cm2 greater than 0.2µm, which is better performance than the particle specification for the system without process (0.07 p/cm2 for particles greater than 0.3 µm). Excursions from the trend reflect the start up of the target and routine change of wafer holder tabs.

In a separate evaluation, the data in Figure 7 show particle levels on W-Ti films. The data show little difference between vacuum hot pressed targets and MRC targets. These results are in direct conflict with the results of Dunlop who suggests that vacuum hot pressed targets generate fewer particles than either inert gas hot pressed or hot isostatic pressed material. The authors believe that Dunlop’s data for vacuum hot pressed material represent the particle contribution of the target, while the data for the other consolidation techniques reflect contributions of flaking from shields and the chamber. If so, these data are not a valid comparison of the different target manufacturing processes. The excursions in particle counts shown in Figure 7 occur at intervals of routine maintenance.

CONCLUSIONS

1. The density of W-Ti sputtering targets is confiied by multiple investigators to be a critical parameter affecting the contribution of the target to particles generated during sputtering.
2. Different consolidation techniques can produce different microstructures in W-Ti sputtering targets, yet a number of these techniques are capable of producing a high density target.
3. Care and attention to maintenance and general housekeeping contribute substantially to reducing particulate levels.

BENEFITS

High density W-Ti sputtering targets provide assurance to semiconductor manufacturers that particulates generated directly from the sputtering target are the lowest possible. Low levels of particulates attributable to any source improve yields at the metallization step of the wafer fabrication process. In addition to improved particle performance, higher sputtering target densities promote faster.pump down of the target to vacuum, and reduce the likelihood of contamination from ambient air.

[1] Waterman, Dunlop and Brat, Proceedings: 7th International IEEE VLSI Multilevel Interconnection Conference, June 1990, p. 329.

[2] Dunlop, Waterman and Brat, J. Vac Sci Tech, AlO, 1991.

[3] Wickersham, Poole and Mueller, Proceedings: 8th International IEEE VLSI Multilevel Interconnection Conference, June 1991, p. 82.

[4] “ECLIPSE”” and **“ECLIPSE-Star™”** are registered trademarks of Materials Research Corporation.

[5] Udler, Marx and Murphy, **Tungsten-Titanium Sputtering Target Technology, MRC** Technical Note #1258,1990.

[6] **Binary Alloy** Phase **Diagrams-Vol.** 3, ed. T. B. Massalski, ASM, Metals Park, Ohio, (1986). p. 2136.

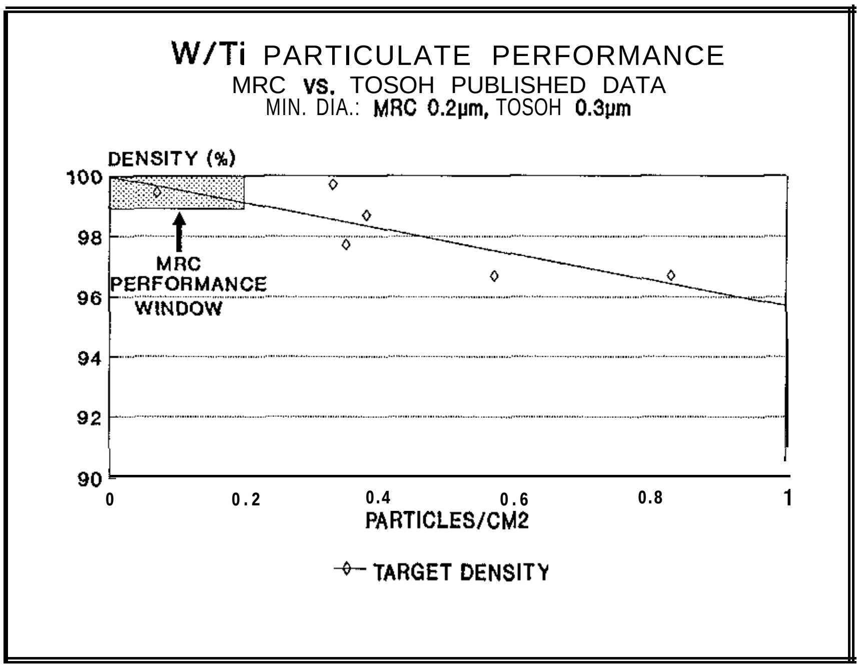

Figure 1: Particle data compared with target density.

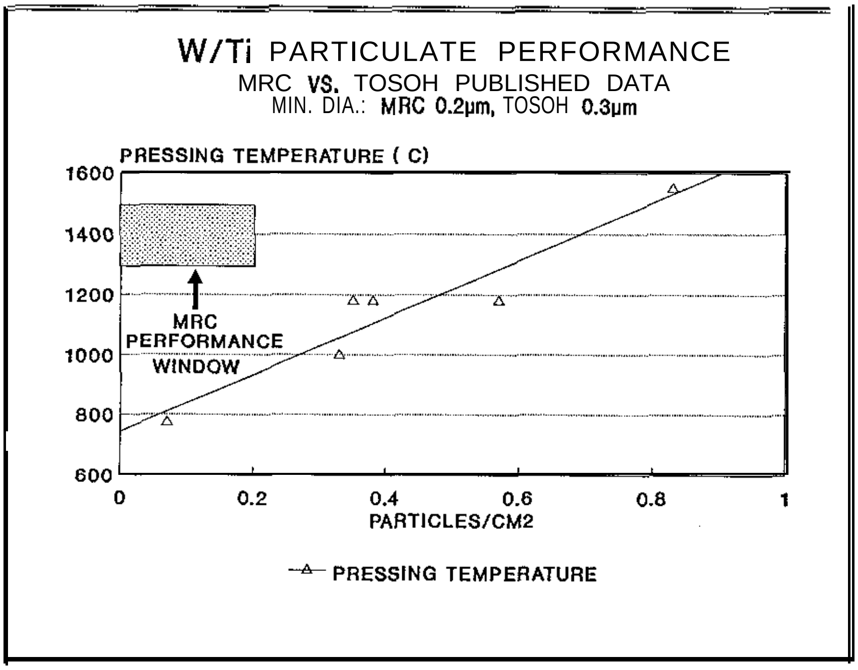

Figure 2: Particle data compared with pressing temperature.

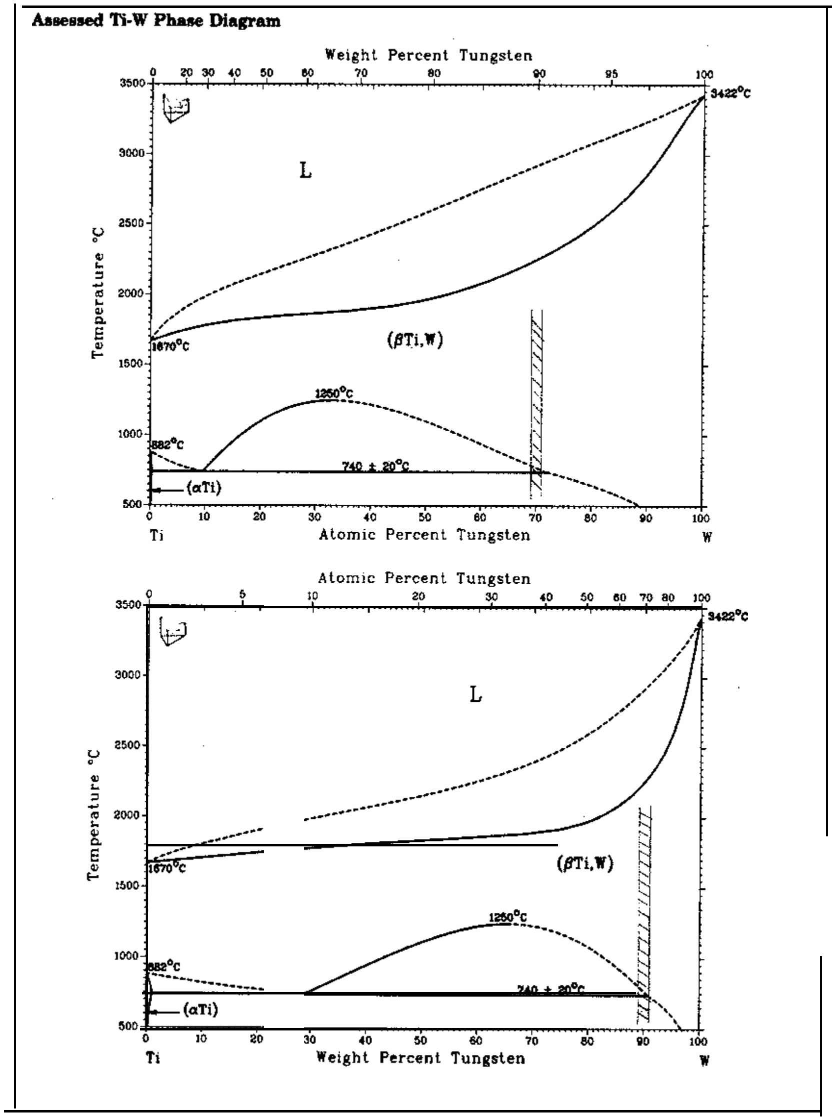

Figure 3: W-Ti phase diagram from <u>Binary Alloy Phase Diagrams</u>, Vol. 3[5].

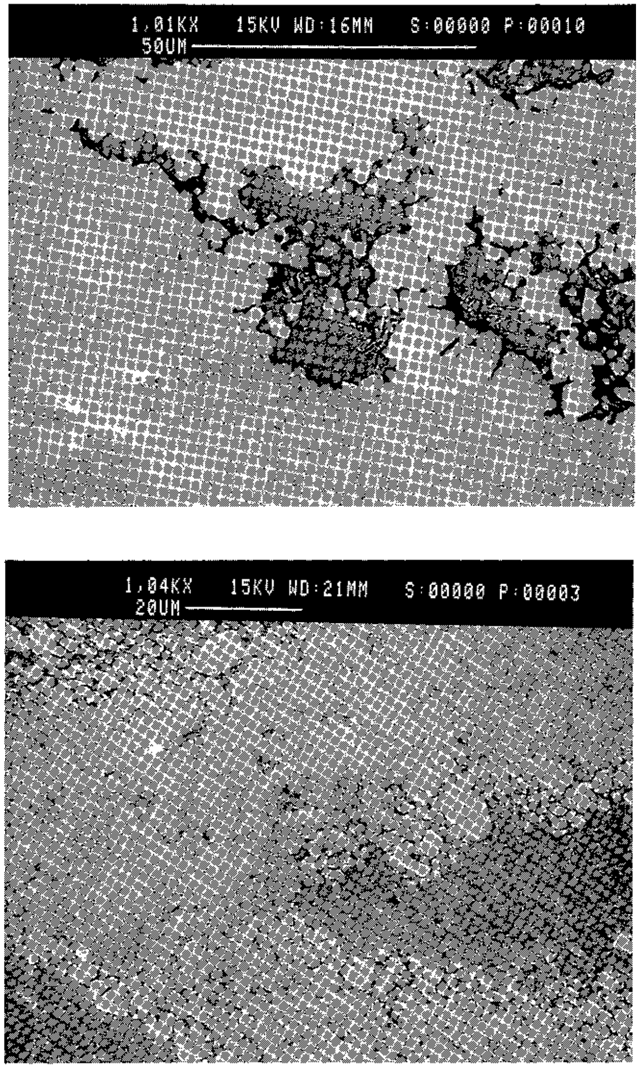

Figure 4: Microstructure of W-Ti sputtering targets.
a) MRC Eclipse™.
b) vacuum hot pressed.

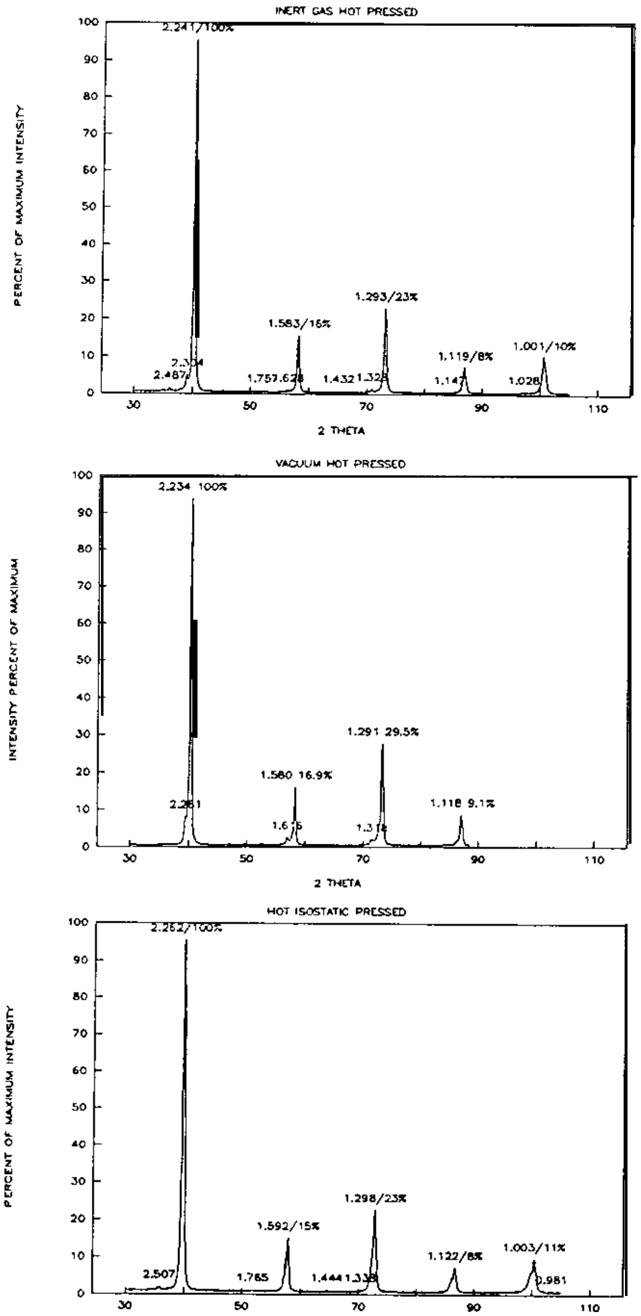

Figure 5: X-ray diiraction data of W-Ti sputtering targets.

a) MRC Eclipse™.
b) vacuum hot pressed.
c) hot isostatic pressed.

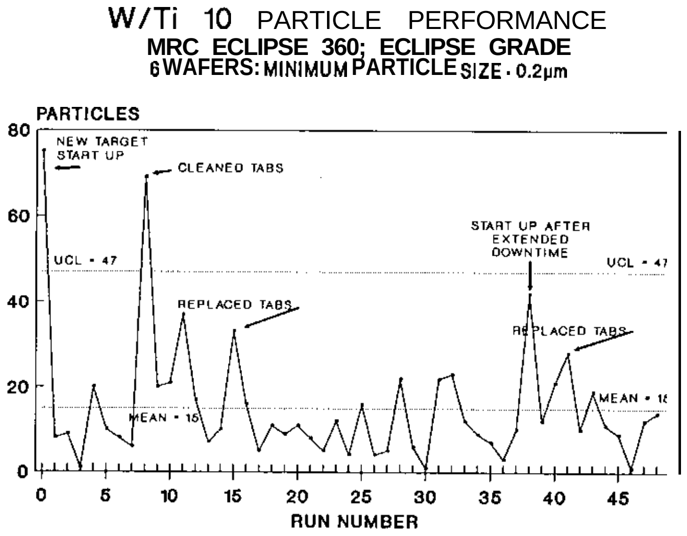

Figure 6: MRC particle performance under “walk with process gas” conditions for a 150mm wafer application.

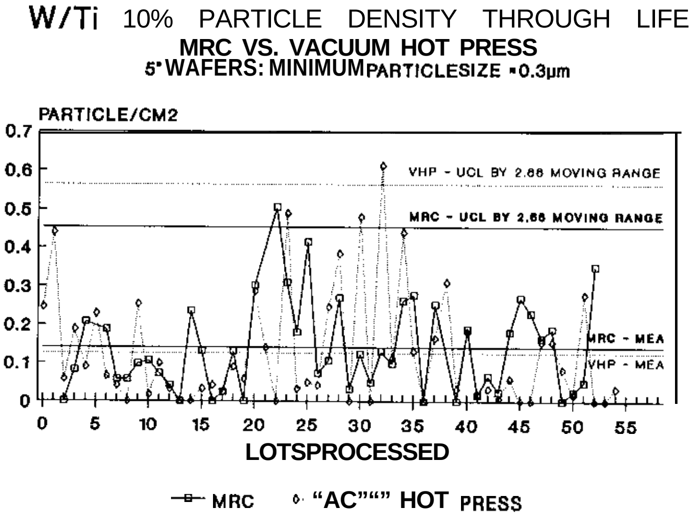

Figure 7: MRC particle performance for W-Ti films on a 125mm wafer application. Two sets of data are shown: an MRC target and a vacuum hot pressed target.

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