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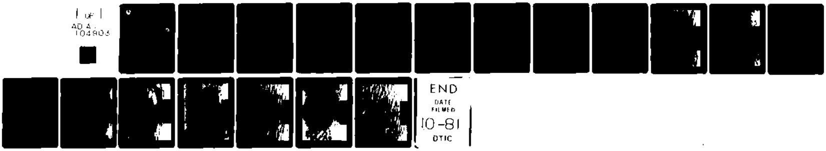

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RESEARCH AND DEVELOPMENT TECHNICAL REPORT

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FURTHER INVESTIGATION OF ETCHANTS FOR CHEMICALLY POLISHING SC-CUT QUARTZ CRYSTALS

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SEPTEMBER 1981

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A series of solutions were investigated for chemically polishing SC-cut quartz crystals. Solutions of concentrations

(1) $\frac{H_2O}{HF} = \frac{2}{1}$, (2) $\frac{H_2O}{HF} = \frac{3}{2}$, (3) $\frac{H_2O}{HF} = \frac{1}{1}$, (4) $\frac{H_2O}{HF}$ = [illegible], and [illegible]

were prepared. It was determined that some of these solutions can chemically polish SC-cut crystals in less time than solutions previously reported.

DD FORM 1473, 1 JAN 73 — EDITION OF 1 NOV 65 IS OBSOLETE

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered) etching time minimizes the problem of water evaporation from the etching bath. Also, the new solutions are easier to prepare than those employing NH_4F.

CONTENTS

Introduction

The results of experiments aimed at finding a chemical po[illegible] cut quartz crystal plates have been reported previously[1]. Some of the solutions evaluated in the previous experiments did not produce a chemically polished surface on either face of the crystal, some produced [illegible] polish on one side of the crystal and not the other, and some were able to polish both sides of the crystal. It was shown that an excellent chemical polish could be obtained for both sides of an SC-cut crystal with a solution of NH_4F (40%) : HF (49%) = 4:1. About two hours of etching time was required at 75°C to etch $\Delta f = 15\ f_o f_f$*. It was also shown that a solution of H_2O : HF (49%) = 4 : 1 could chemically polish SC-cut crystals [illegible] two and one half hours at 75°C to the same depth of etch. The purpose [illegible] the work described in this report was to investigate additional solutions in the H_2O : HF (49%) series to determine if other such solutions are capable of chemically polishing SC-cut crystals. SC-cut crystals have the potential for providing improved crystal resonators for applications in navigation, communications and identification systems.

Etching Experiments

Five solutions were prepared for this study as follows:

(1) H_2O : HF = 2 : 1 (2) H_2O : HF = 3 : 2 (3) H_2O : HF = 1 : 1

(4) H_2O : HF = 2 : 3 (5) H_2O : HF = 1 : 2

The HF concentrations were not measured; the 49% specified by the manufacturer was assumed to be correct. The calculated concentrations based on these mixtures are shown in Table 1. The SC-cut crystals [illegible] natural quartz and had nominal angles of $\phi = 21^{\circ}\ 56' \pm 20'$ and $\theta = 34^{\circ}$ [illegible] $\pm 5'$. The diameters were 14 mm and the blanks had an initial frequency [illegible] 4.060 MHz. The crystals were plano-plano and had 1 μm lapped surfaces. Before chemical polishing, the crystals were cleaned thoroughly.

The etching and chemical polishing experiments were [illegible] 75°C and, in one instance, at 70°C. Table 1 shows the [illegible] inspection of both sides of the crystals etched in the [illegible] solutions. A polished surface was obtained on both sides [illegible] except those etched in the H_2O : HF = 1 : 2 solution. Crystals from [illegible] category were then surface profiled using a Tencor Alpha-step profilometer. An estimate of the surface roughness for each crystal [illegible] Alpha-step measurements is shown in Table 2. [illegible] were estimated by calculating the root mean square deviation [illegible] imaginary center line through the Alpha-step profile wh[illegible] that the areas under the profile above and below the line were approximately equal, as estimated visually. The values of surface [illegible] obtained are shown in Table 2.

*fo = initial frequency in MHz, f_f = final frequency in [illegible]

Polished crystals obtained from each of the etching solutions investigated were also examined by scanning electron microscopy. Electron micrographs are shown in figures 1 thru 10 for both sides of each crystal. It can be seen from the electron micrographs that there is a difference in surface roughness between the two sides of the crystals. The difference is more pronounced for the crystals etched in H_2O : HF = 1 : 1 based on both the electron micrograph and Alpha-step results. (When tested with an electrometer, the "rough" sides are positive on compression[2].)

To assure that the surfaces are etched evenly, it is important to remove all contaminants which may be impervious to the etchants[3]. It should be noted that Ward[4] has found that the NH_4F : HF = 4 : 1 etching solution described previously is more forgiving of surface contamination for SC-cut crystals than the H_2O : HF = 2 : 1 solution.

Conclusion

SC-cut quartz plates can be chemically polished on both sides with the designated solutions, at least up to concentrations of H_2O : HF = 1 : 1. Starting with 1 μm lapped surfaces, surface roughnesses of approximately 0.05 μm and 0.04 μm can be achieved for the two sides after etching to $\Delta f = 15\ f_o f_f$. These solutions provide a faster etching rate than those previously reported. For example, a solution of H_2O : HF = 1 : 1 at 75^o can chemically polish an SC-cut plate to a depth of $\Delta f = 15\ f_o f_f$ in 42 minutes compared to about two hours for NH_4F : HF = 4 : 1 at the same temperature.

Acknowledgements

The authors gratefully acknowledge the contributions of D. Eckart for preparing the SEM micrographs and F. Ivins for providing the Alpha-step profiles.

References

1. J.R. Vig, R.J. Brandmayr and R.L. Filler, "Etching Studies on Singly and Doubly Rotated Quartz Plates," Proc. 33rd ASFC, pp. 351 - 358 (1979); Copies available from Electronic Industries Assoc., 2001 Eye Street, N.W. Washington, D.C. 20006.

2. John G. Gualtieri, "A Simple Method for Location of the Mounting Positions for Low Acceleration Sensitivity SC-cut Resonators," Technical Report DELET-TR-81-5, ERADCOM, Fort Monmouth, NJ, Feb 1981.

3 J.R. Vig, J.W. LeBus and R.L. Filler, "Chemically Polished Quartz," Proceedings of the 31st ASFC, pp. 131 - 143, (1977); Copies available from Electronic Industries Assoc., 2001 Eye Street, N.W. Washington, D.C. 20006.

4. R.W. Ward, Colorado Crystal Corp, Private Communication, Jan 1981, also, to be published in the Proceedings of the 35th ASFC, 1981.

TABLE 1

ETCHING RATES

ETCHANT	SIDE 1/SIDE 2	TEMPERATURE °C	TIME OF ETCH (min)	$\frac{\Delta f}{f_o f_f t}$
$\frac{H_2O}{HF} = \frac{2}{1}$ (16% HF)	P/P	75	73	0.22
$\frac{H_2O}{HF} = \frac{3}{2}$ (20% HF)	P/P	75	49	0.32
$\frac{H_2O}{HF} = \frac{1}{1}$ (24% HF)	P/P	75	42	0.36
$\frac{H_2O}{HF} = \frac{2}{3}$ (29% HF)	P/P	75	30	0.51
$\frac{H_2O}{HF} = \frac{1}{2}$ (33% HF)	P/R	70	30	0.68

P = Polished

R = Rough

f_o = Initial Frequency in MHz

f_f = Final Frequency in MHz

Δf = $(f_f - f_o)\ 10^3$ in KHz

t = Time in min

TABLE 2

SURFACE ROUGHNESS OF SC-CUT QUARTZ CRYSTALS ($\frac{\Delta f}{f_o f_f}$ = 15)

ETCHANT	VERTICAL MAGNIFICATION	ESTIMATED ROUGHNESS (μm)	SIDE
$\frac{H_2O}{HF} = \frac{2}{1}$	100,000X	± 0.05	1
Same	100,000X	± 0.05	2
$\frac{H_2O}{HF} = \frac{3}{2}$	100,000X	± 0.05	1
Same	100,000X	± 0.04	2
$\frac{H_2O}{HF} = \frac{1}{1}$	100,000X	± 0.04	1
Same	100,000X	± 0.04	2
$\frac{H_2O}{HF} = \frac{2}{3}$	100,000X	± 0.05	1
Same	100,000X	± 0.04	2
$\frac{H_2O}{HF} = \frac{1}{2}$	100,000X	± 0.04	1
Same	100,000X	± 0.09	2

* Measured with a TENCOR INSTRUMENTS Alpha-Step profile meter
Horizontal Magnification: 50X
Distance measured: 6mm

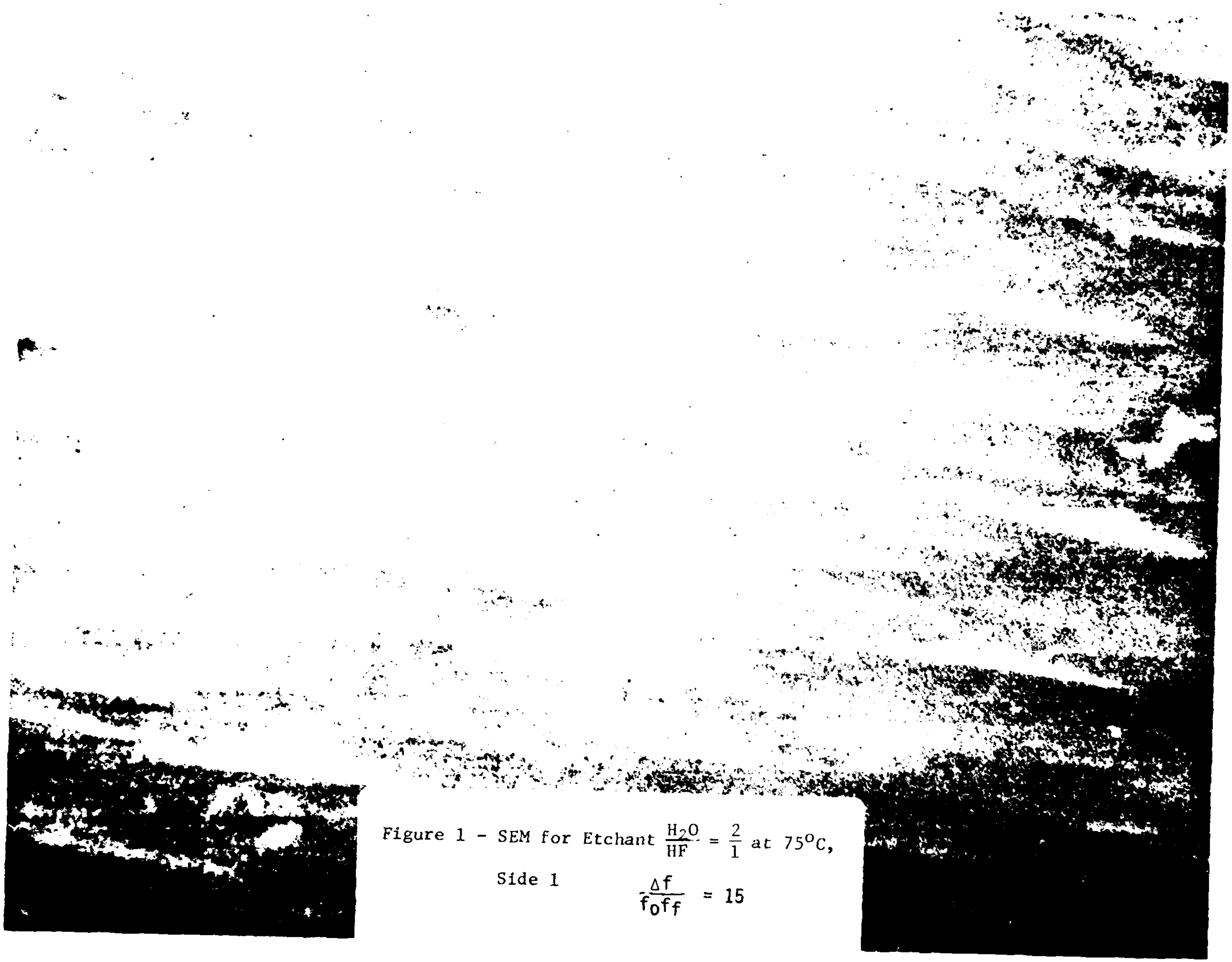

Figure 1 - SEM for Etchant $\frac{H_2O}{HF} = \frac{2}{1}$ at 75°C,

Side 1 $\frac{\Delta f}{f_0 f_f} = 15$

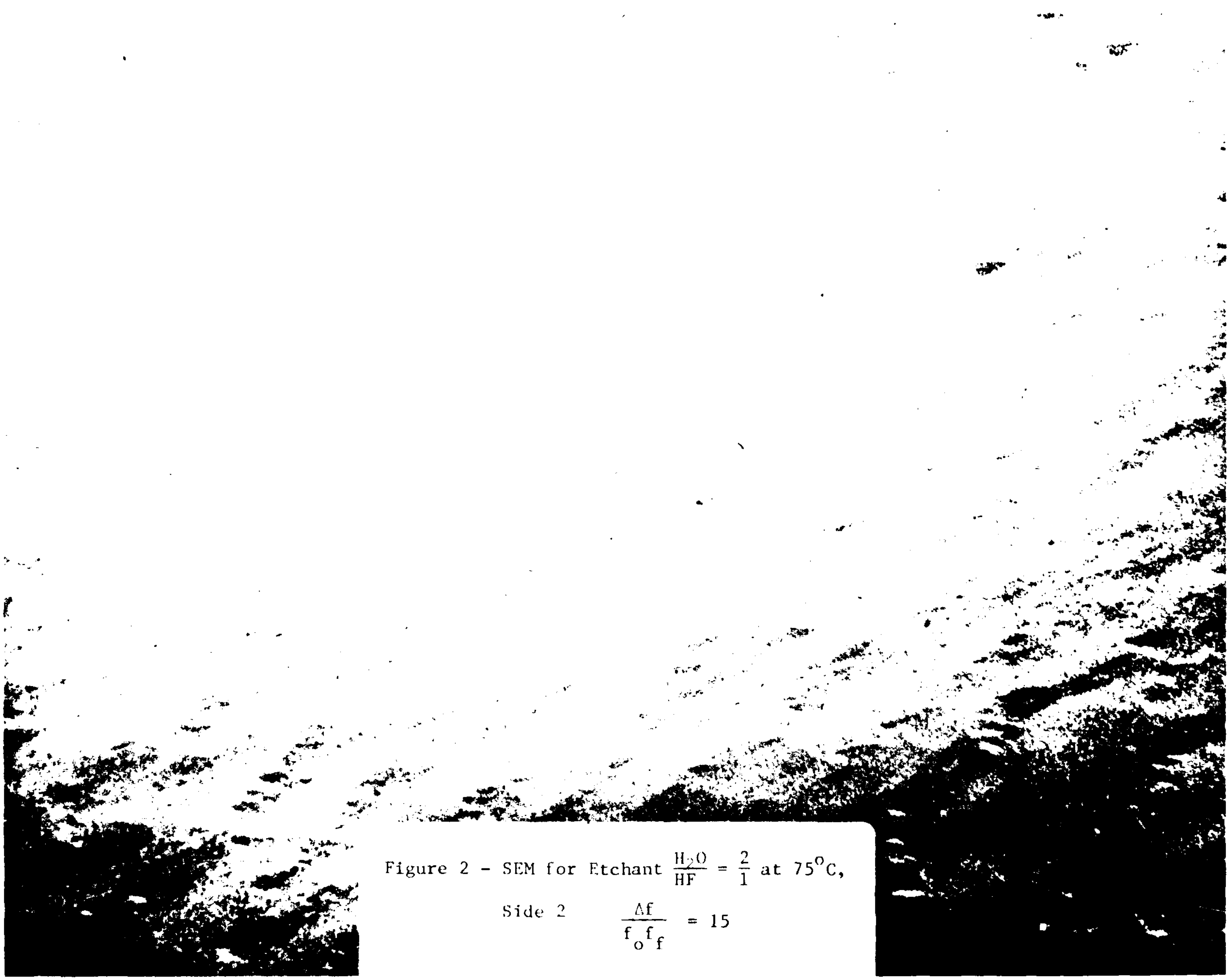

Figure 2 - SEM for Etchant $\frac{H_2O}{HF} = \frac{2}{1}$ at 75^oC,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$

Figure 3 - SEM for Etchant $\frac{H_2O}{HF} = \frac{3}{2}$ at 75^oC,

Side 1 $\frac{\Delta f}{f_o f_f} = 15$

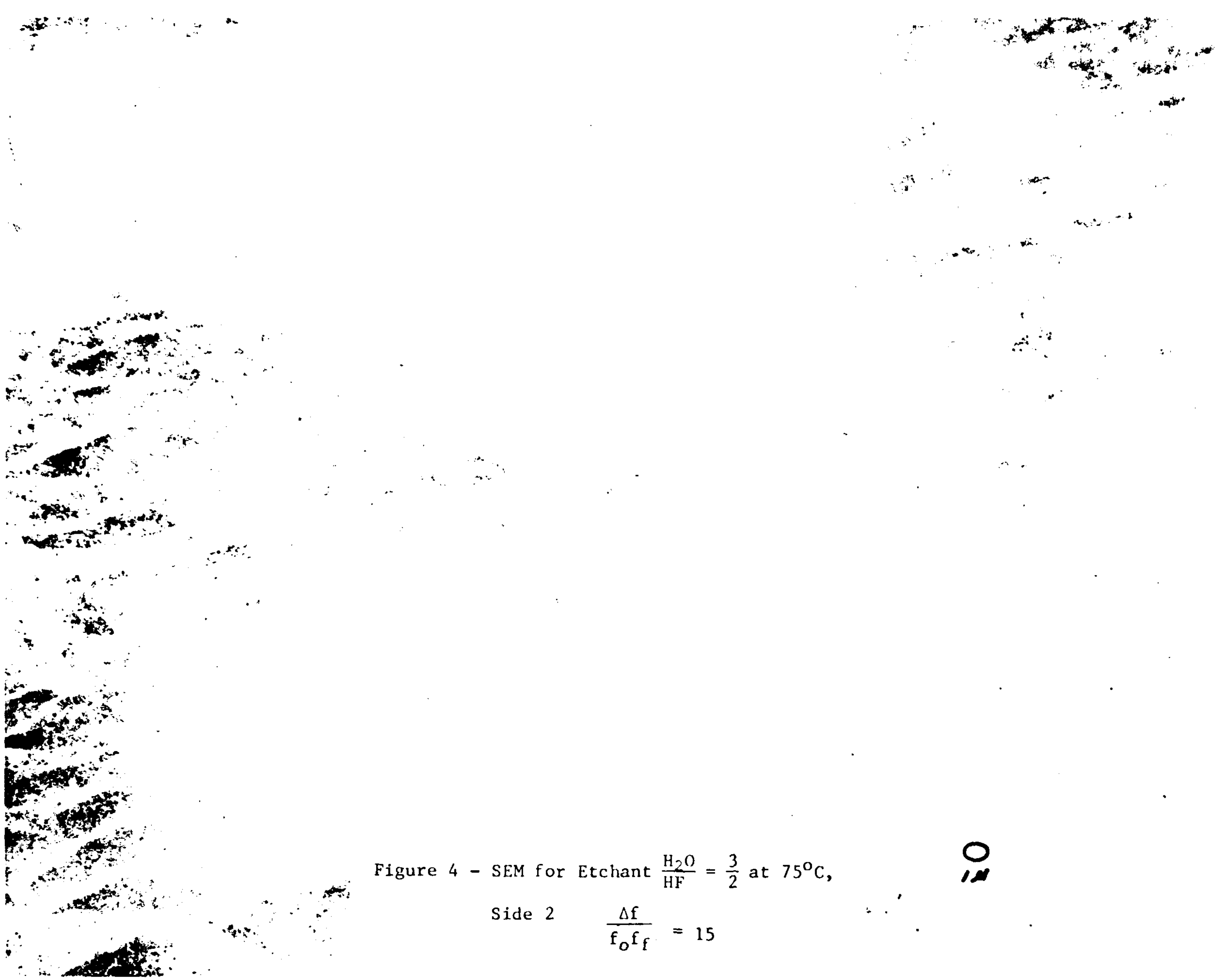

Figure 4 - SEM for Etchant $\frac{H_2O}{HF} = \frac{3}{2}$ at 75°C,

Side 2 $\quad \frac{\Delta f}{f_o f_f} = 15$

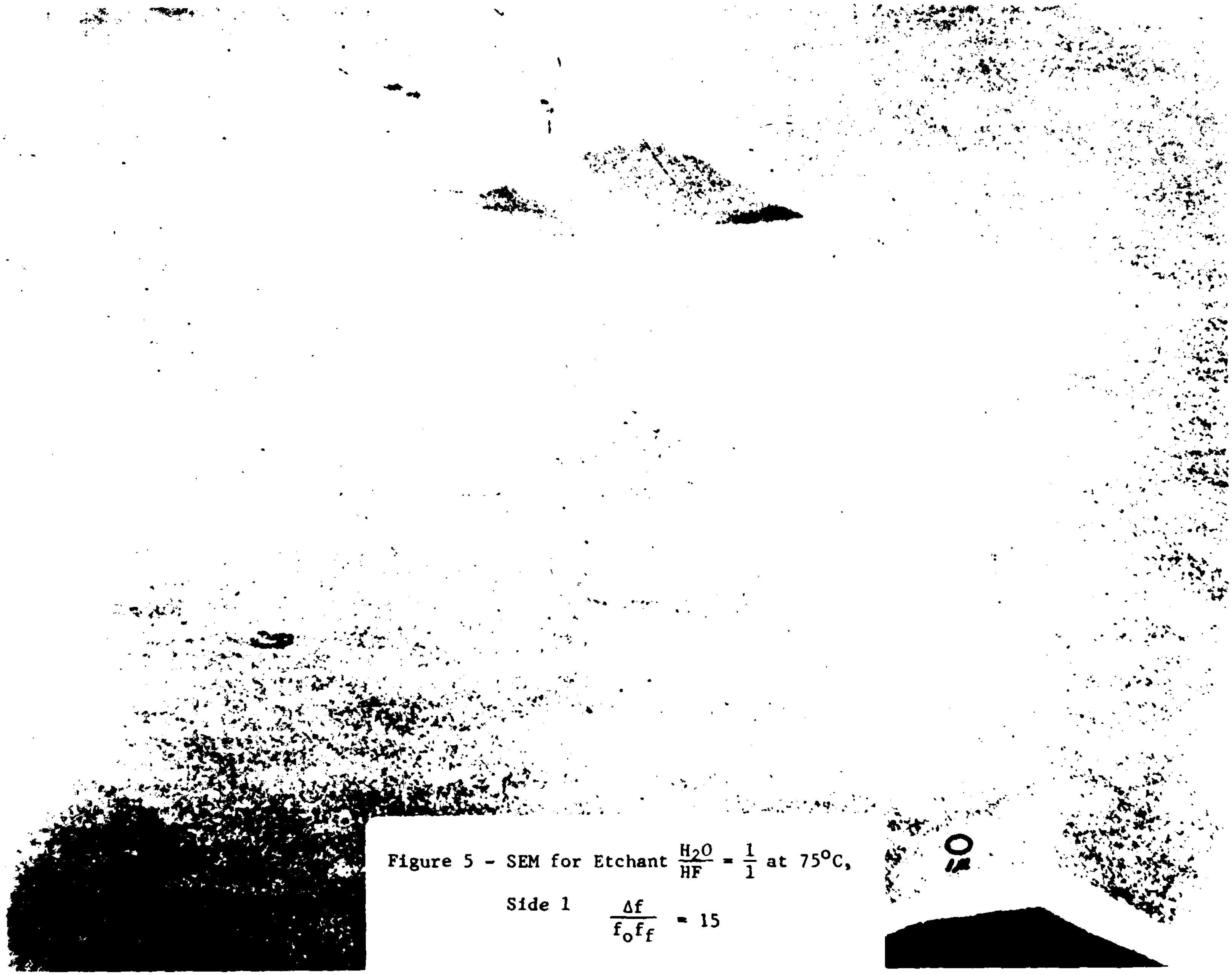

Figure 5 - SEM for Etchant $\frac{H_2O}{HF} = \frac{1}{1}$ at 75^oC,

Side 1 $\frac{\Delta f}{f_o f_f} = 15$

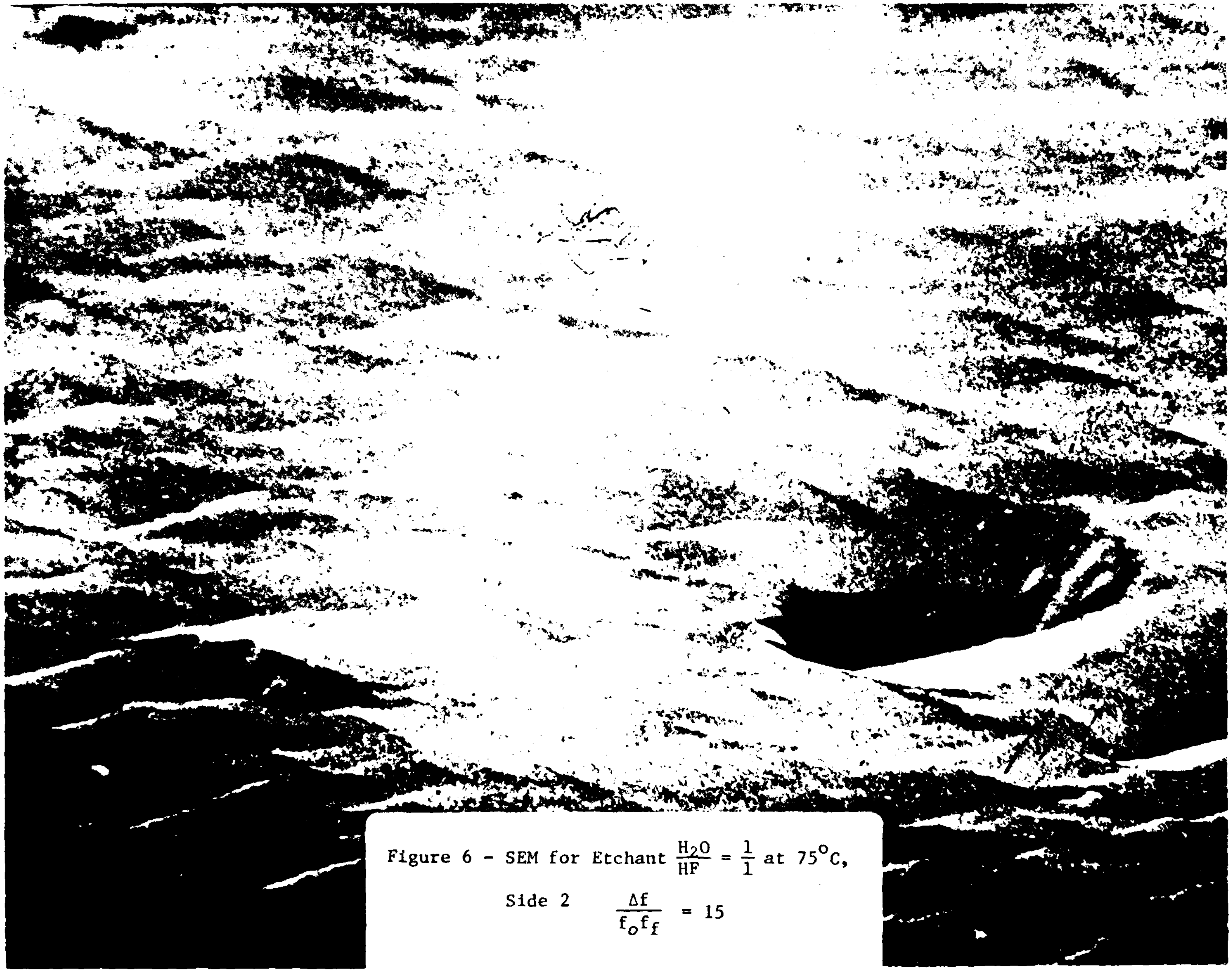

Figure 6 - SEM for Etchant $\frac{H_2O}{HF} = \frac{1}{1}$ at $75^{o}C$,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$

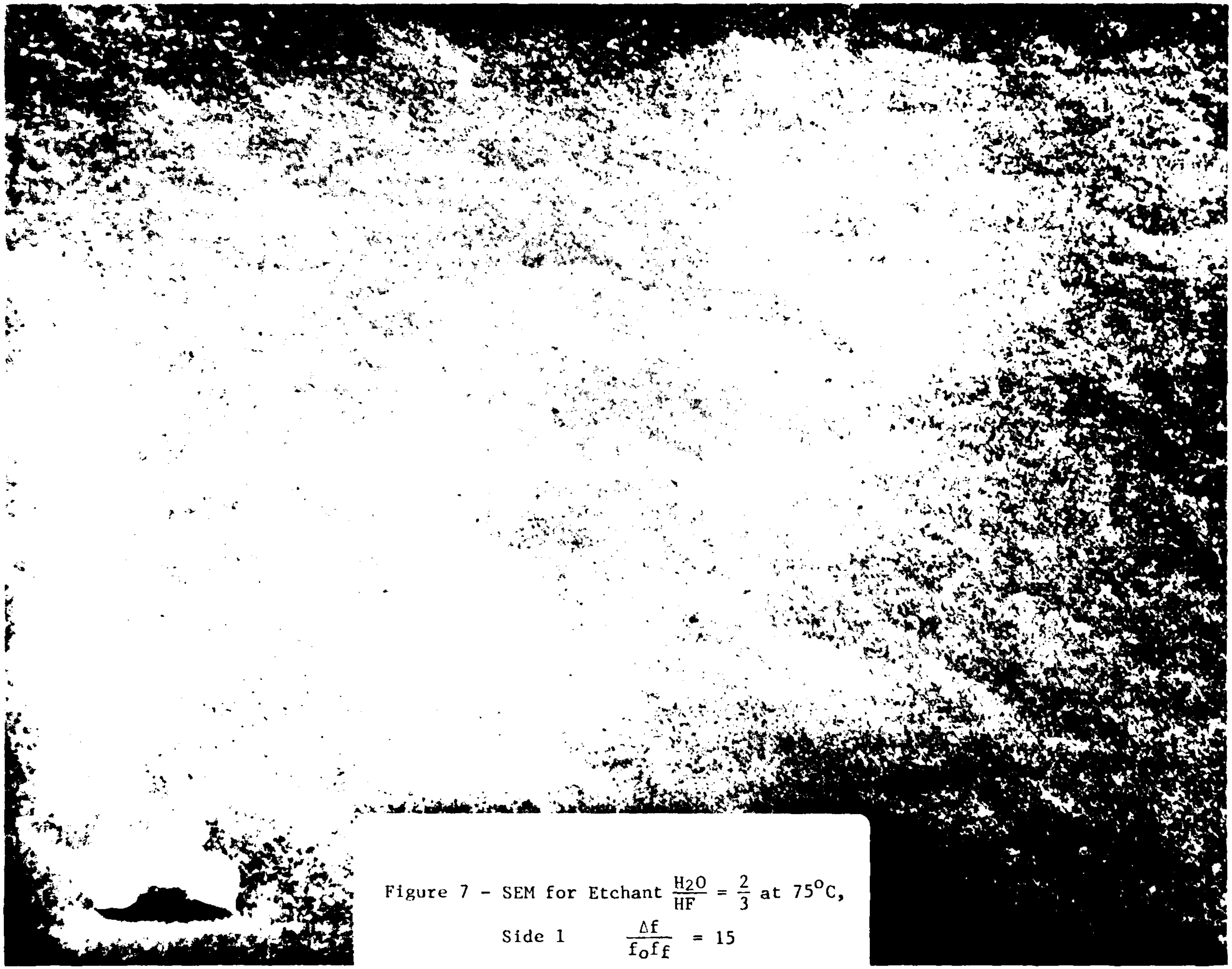

Figure 7 - SEM for Etchant $\frac{H_2O}{HF} = \frac{2}{3}$ at 75^oC,

Side 1 $\frac{\Delta f}{f_o f_f} = 15$

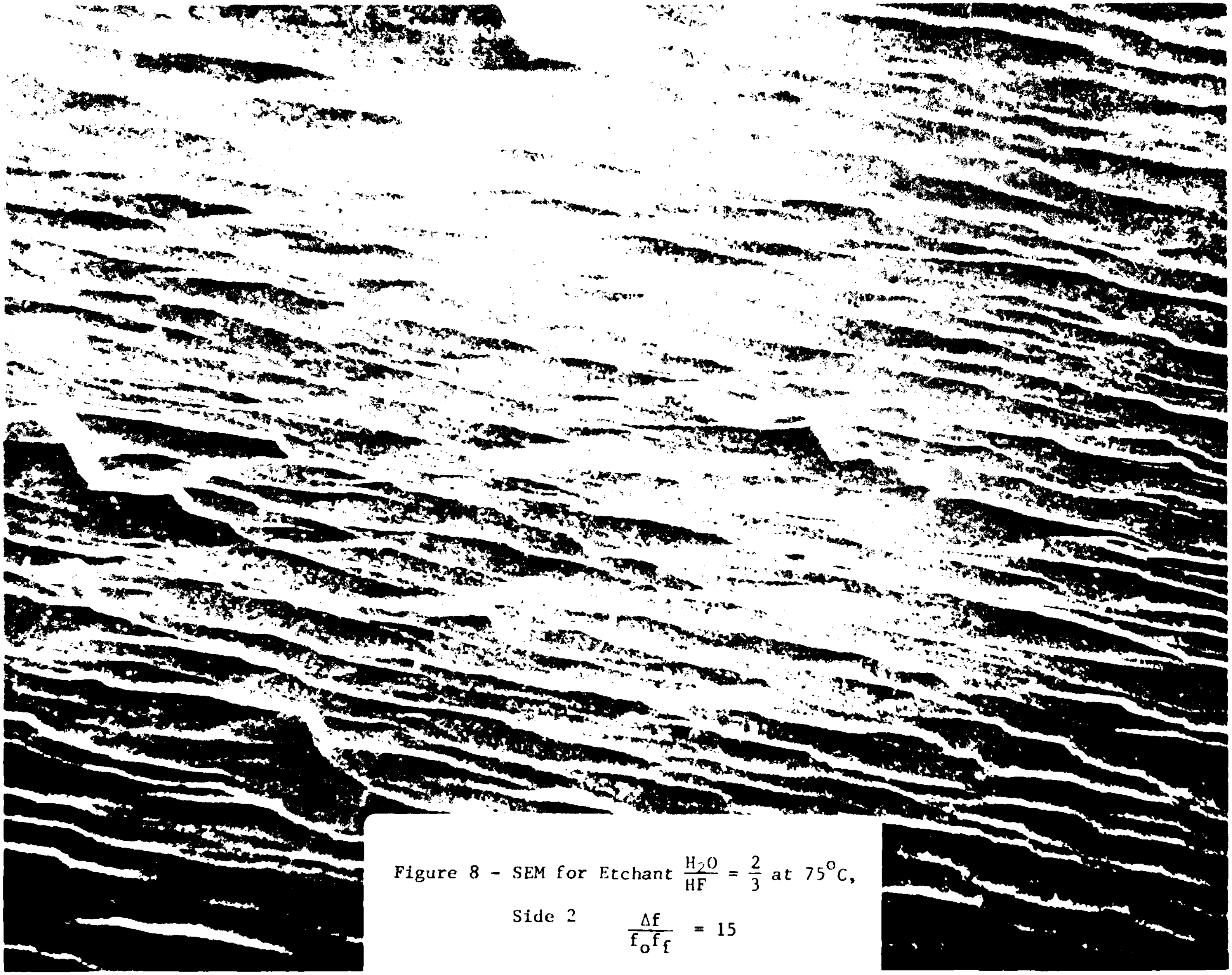

Figure 8 - SEM for Etchant $\frac{H_2O}{HF} = \frac{2}{3}$ at 75^oC,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$

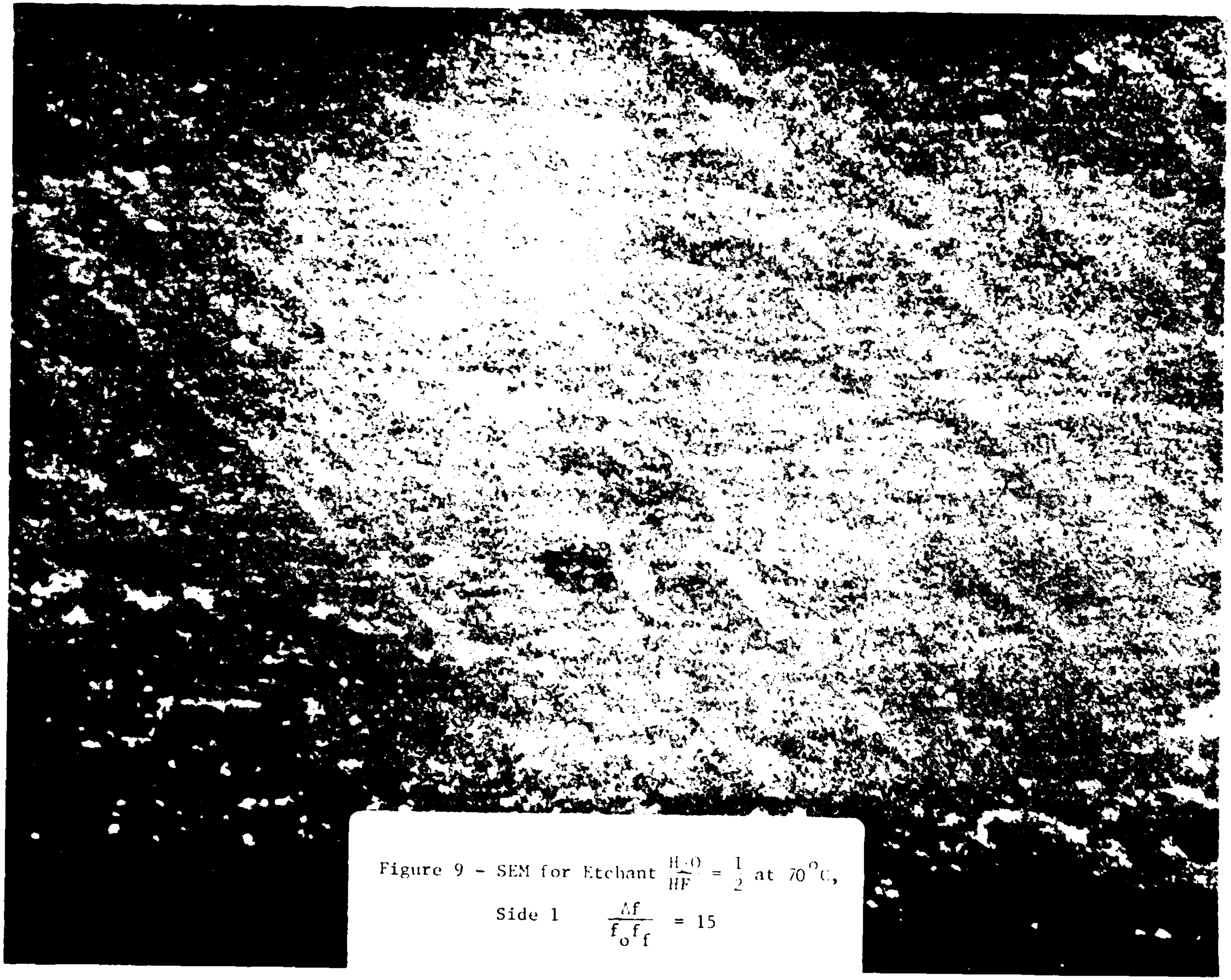

Figure 9 - SEM for Etchant $\frac{H_2O}{HF} = \frac{1}{2}$ at $70^{\circ}C$,

Side 1 $\quad \frac{\Delta f}{f_o f_f} = 15$

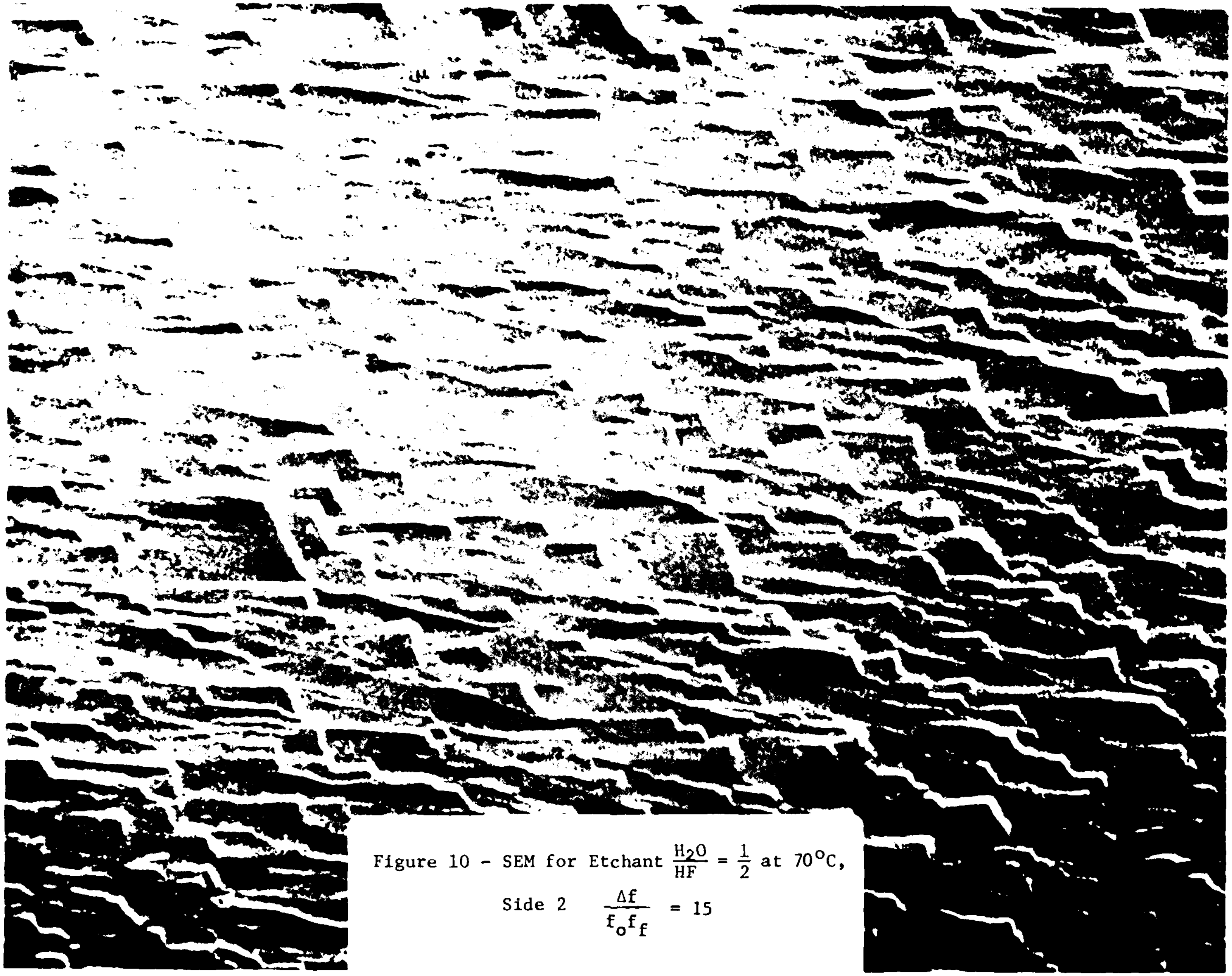

Figure 10 - SEM for Etchant $\frac{H_2O}{HF} = \frac{1}{2}$ at $70^{o}C$,

Side 2 $\frac{\Delta f}{f_o f_f} = 15$