

In the SEM all samples were run in triplicate on two areas. The operating conditions were: 100 s fixed time, 25 kV, 50× magnification. By X-ray fluorescence, in which a larger area of the specimen is viewed by the X-ray beam, most of the results represent one area viewed for 100 s. In the cases of both SEM and X-ray fluorescence the results are presented as the net integrated number of counts (total counts minus background counts) of aluminum  $K\alpha$ , 1.49 keV, per 1000 s. A tungsten tube operated at 10 kV was used as the source in the X-ray fluorescence experiment.

Inspection of the data indicates fairly good correlation of the SOAP 340, 125, and 35 ppm aluminum samples with either the SEM/ED or the X-ray fluorescence data. The 71 ppm aluminum sample appears to be off in both cases, which could be due to some irregularity in sampling, sample preparation, or to an incorrect aluminum analysis. The correlation below 35 ppm aluminum in Table 2 is poor. This is the result of a poorer signal-to-noise ratio—the lower aluminum values do not produce sufficient counts above background to give good statistics. However, this situation can be substantially improved by using more sample, which will increase the aluminum count and not change the background appreciably.

Quantitative analysis by either SEM or X-ray fluorescence can also be improved by comparing the data against a set of standards and by using the ratio of the element count vs some internal standard—very much the way it is done in spectrochemical analysis. X-ray spectroscopy offers the advantage that all elements, not only certain selected elements as in SOAP, are displayed simultaneously.

## DISCUSSION

### L. Leonard<sup>3</sup>

It would be appreciated if the authors would provide some clarification concerning several aspects of their experimental techniques and also replies to several comments on their investigation.

Was the maximum size of the particulate matter in the oil samples limited by an in-line filter in the hydraulic system? If so, what was the nominal filter size employed? This is of great importance in wear monitoring since wear modes such as spalling or excessive abrasion or cutting wear generate primary particles well in excess of 5 microns. Accordingly, if fine in-line filters are used in order to foster longer component life by eliminating both internally and externally generated debris, the validity of monitoring the oil by SOAP or SEM techniques becomes questionable.

The authors indicate in their abstract that they found a good correlation between the SOAP and SEM analyses and the actual condition of the wearing components. However, in the paper itself there is no discussion of a correlation between the surfaces of the components and the morphology of the particulate matter observed in the SEM. Since there are substantial data in the literature concerning the correlation of particle morphology with wear modes, it would be of interest to learn to which wear modes the authors attribute the particles that they observed.

The authors' findings that the silicon containing particles larger than 5 microns were apparently not appropriately rated by SOAP analysis is consistent with the observations of other investigators. In the on-going Oil Analysis Program sponsored by the Naval Air Engineering Center, Lakehurst, N. J., test bearing and gear failures could not be predicted on the basis of SOAP analysis. While this technique showed little or no increase in iron, direct counting of the particles filtered from the oil did determine significant increases in the numbers of particles larger than 5 microns prior to and after failure.

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### 6 Summary

Good qualitative correlation was obtained between aluminum analysis by SOAP and X-ray spectroscopy with SEM. Preliminary quantitative correlation attempts are encouraging, and better correlations should be established with more experiments. The X-ray spectroscopy methods, like SOAP, show other elements present in the oils. Correlation of SOAP to analyses of other particles present in the oil was poor—and future work is needed to determine the influence of particle size or other factors on spectroscopical analysis of elements such as silicon, calcium, etc.

### 7 Acknowledgment

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### References

- 1 "Spectrometric Oil Analysis," in Department of the Army Technical Bulletin, TB 55-6650-300-15, 5 Aug. 1970, and "Maintenance of Supplies and Equipment, Spectrometric Oil Analysis," in Army Regulation No. 750-13, 1 Nov. 1970.
- 2 Johari, O., "Characterizing Materials with the SEM Today," *Research Development*, Vol. 25 (8), Aug. 1974, pp. 16-23.
- 3 Johari, O., and DeNec, P. B., "Handling, Mounting and Examination of Particles for SEM," in *Scanning Electron Microscopy/1972*, IIT Research Institute, Chicago, Ill., pp. 249-256.

As a final question, since the authors indicate that most of the aluminum particulate matter in their analysis was smaller than 5 microns in size, was there any attempt made to determine how much of this particulate matter was not trapped by the 5 micron filter? This, of course, is very important in comparing the SEM and X-ray fluorescence techniques with the SOAP results.

### R. K. Tessmann<sup>4</sup>

Wear debris analysis is becoming a very important tool in the evaluation of wear processes in fluid systems. The authors of this paper are to be commended for their work in the area. As in any growing area of investigation, several techniques are advanced. This paper presents data derived using some of these methods from oil samples extracted from working systems in an attempt to reveal the degree of correlation which can be achieved. As was pointed out in the paper, good qualitative correlation was shown for aluminum; however, the correlation for other debris particles such as silicon was poor.

The SOAP method of evaluating the wear debris present in the fluid of a hydraulic system has been used with varying degrees of success for a number of years. This technique relies upon an increase in the concentration of one or more primary elements in the wear debris to predict the severity of the wear processes which are occurring. The SEM and X-ray data are capable of providing not only the elemental concentration information but also subjective knowledge concerning the morphology of the wear debris particles. In general, then, it would appear that SEM and X-ray data are much more powerful in evaluating wear processes. Assuming that a correlation was achieved further elaboration by the authors in regard to the significance of the correlation between SOAP and SEM data would put their work in proper perspective.

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## Authors' Closure

We appreciate the comments of Dr. Leonard and Professor Tessmann.

There are filters located on the incoming and outgoing hydraulic lines from the reservoirs, as well as on selected locations in each component. These filters should limit the size of the particulate matter reaching various components. While we were unable to obtain exact nominal sizes for each filter we expected that the particles of 5  $\mu\text{m}$  and up should have been trapped. In another paper (to be published in *Proceedings of the Wear Conference*, St. Louis, April 1977) details of hydraulic system and individual components have been described. As discussed there, entry of particulate matter in lines and components from external sources is possible. These particles may not see the filters and be detected as we have described here.

The part condition data were not included in this paper so as to keep it to a reasonable length. From the other paper (cited above),

the predominant wear mode noted was abrasion of parts caused by hard silica and chrome particles getting embedded in seals.

We have been unable to find literature references indicating relationship of particulate size and its detection by SOAP. Clearly it is an area where systematic investigation is required if the reliability of the use of SOAP is to be increased.

As explained above, exact sizes of filters were not known. Exact correlations between fractions of a given size particle trapped/not trapped by a given filter were not developed.

Our work is a first attempt in this important area which needs to be further investigated. At present stage, SEM and X-ray analysis can supplement SOAP data in several areas, e.g., (1) when the elemental concentration shown by SOAP of any element is high, because here determination of individual particle morphology and chemistry would provide valuable additional information; (2) when a system or a component performs poorly despite good SOAP analyses; and (3) when the significance of SOAP analyses is not clear, etc. As more techniques for rapid examination are developed, the role of SEM should further increase.