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THE COMPLEXITY OF SEM-EDS – WHAT AFFECTS THE QUALITY OF OBTAINED RESULTS?

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Abstract

SEM-EDS is a powerful technique that is constantly being improved and supplemented with other material characterization techniques. However, obtained results might be interpreted in different ways as well as the possibility of poor quality of recording itself. The reasons are numerous: sample preparation, SEM operational parameters (working distance, probe current, process time…), EDS detector type, additional WDS – if it is necessary…etc. but first of all is what is the purpose of analysis and what precision do we need. Consequently, the experience of the operator itself is very important. This paper provides an overview of how individual parameters affect the quality of SEM-EDS results of different samples.

***Keywords**: SEM-EDS, techniques, operational parameters, samples.*

1. INTRODUCTION

Materials characterization is complex and consists of different types of methods and techniques. Among them is SEM-EDS which is very detailed and varied technique. We can use it for various kinds of samples – from mineral to biological, and for various purposes – industrial, scientific, educational, medicine etc. SEM-EDS nowadays is a modern, very usable and always upgradeable, which can be supported by constantly improved detectors, software and following contents.However, despite the constant progress of the device, new questions arise in the very interpretation of the results. These questions are sometimes simple, but the answers are not so because there are too many factors that influence on.

Before starting the SEM-EDS you have to know which kind of analysis you want. If you really need to know the composition of your sample with an error of the 1-2% element by element, you should use quantitative analysis with standards. If you are just interested in recognizing different phases, than a semi-quantitative analysis will be enough. Furthermore, you need to consider using WDS instead EDS if it is necessary. The WDS spectrometer can measure trace elements and it can be used as additional to EDS if there is overlapping peaks. That not means that WDS is the better solution and that you can use it rather than EDS. In practice, it's better to use EDS for unknown sample because major elements will be rapidly identified, but in all cases the final analysis depends on operator itself [1-3].

There are several differences in EDS detectors that could be important for analyses. One is energy resolution – if it is better, than it can be use for prevent overlapping peaks. Sensor size is also important – if you want high quality maps, than you have to use larger sensor (50-150 mm^2). Nowadays, SDD detectors are in practice after the Silicon Lithium once, but there are always experienced operators who will give you the best results with the less modern equipment. It doesn't mean that if you have the latest equipment that you will have the best results or quality. It only means that you will have the best opportunity to perform fast analysis, but the quality and reliability will depend of something else.

Sample preparation is also very important and can influence on the quality [4]. The sample has to be clean, dry, non-magnetic and flat if it's possible. It depends of the sample nature itself whether you will use high or low vacuum mode, but there are some facts that you have to respect. Sample preparation varies whether it is conductive or non-conductive. Non-conductive samples must be coated(with gold - the best variant). But, those samples can't be analyzed on gold composition because of accuracy of the analysis. Also, there are samples on which it is not allowed to make any changes (not even coating), such as archaeological samples, jewelry, collectibles, conservation samples, etc.In that case, it is necessary to use low vacuum. At low vacuum and low voltage, it is possible to take a picture to some extent without the occurrence of charging.

2. EXPERIMENTAL

Before recording the sample, depending on the type of sample and the desired image, the starting conditions should be set:

- vacuum mode (HV or LV);
- voltage and probe current;
- signal type (SED, BED or both types in the same time as possible).

As an example of different types of SEM-EDS analysis which are depend of samples and starting conditions, a lot of samples were recorded and analyzed in order to get the best results. These analyzes were done on JEOL JSM IT-300LV.

3. RESULTS AND DISCUSSION

The results shown in Figures 1 – 4 were obtained under different conditions, while the signal type was the same - SED. In Figure 1 SEM snapshots of paper are shown.Both snapshots are obtained at the same probe current (60) and WD≈11. Paper sample was coated as non-conducted sample, as well as powder samples given in Figures 2 - 4.

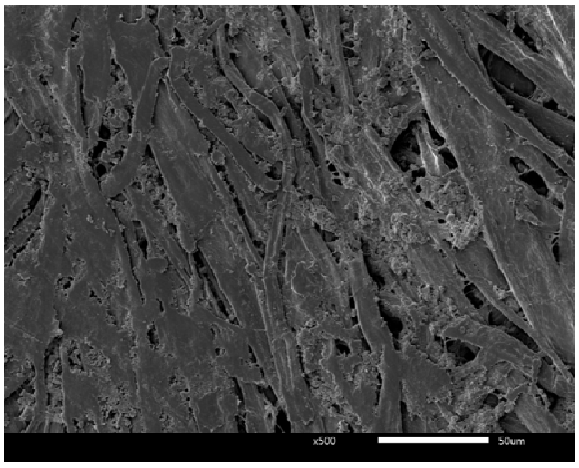

Figure 1a. SEM snapshot of paper, 5 kV

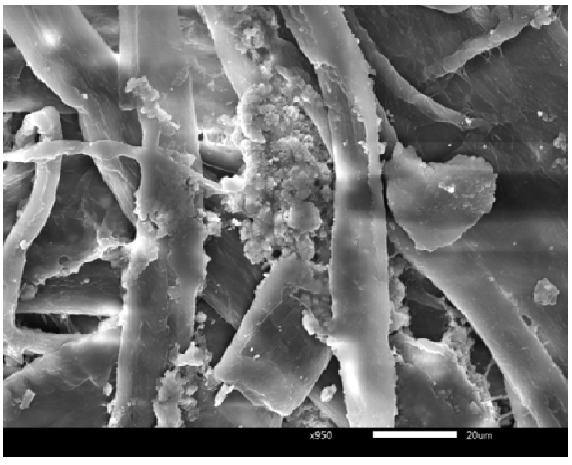

Figure 1b. SEM snapshot of paper, 20 kV

In the Figure 2 the difference between two SEM snapshots of powder, at different WD is given. With smaller WD (Figure 2a) SEM snapshot is very clear and sharp. Taking into account other factors such as kV, PC, magnification etc. (the same for Figures 2a and 2b), it might be concluded that if we want better SEM regardless of the EDS, we can decrease WD.

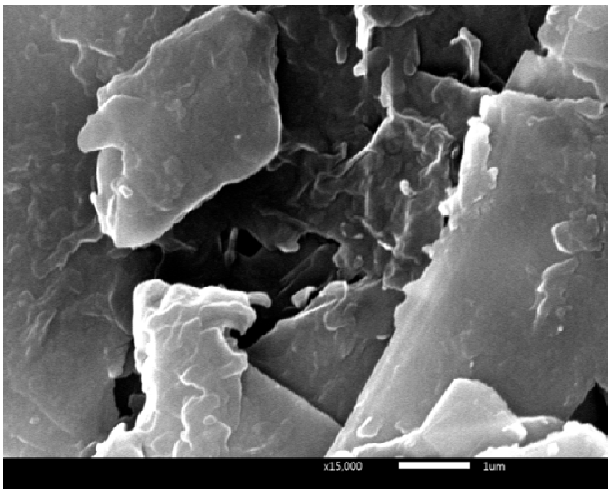

Figure 2a. SEM snapshot of powder
20 kV, PC 40, WD 7.6, M 15000

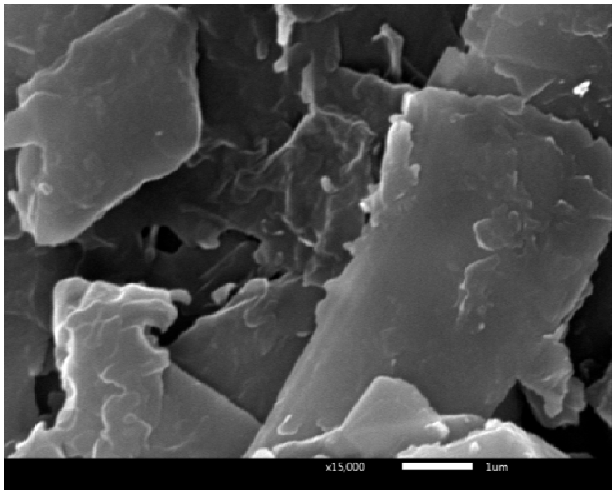

Figure 2b. SEM snapshot of powder
20 kV, PC 40, WD 11, M 15000

Unlike Figure 2, the next example shows the difference between snapshots with different probe current (Figure 3). Figure 3b in comparison with Figure 2a is with poor quality. If the voltage increased at 30 kV at the same conditions as given in Figure 2a, the details in the photo will be clearer, but the structure of the sample will be lost (Figure 4).

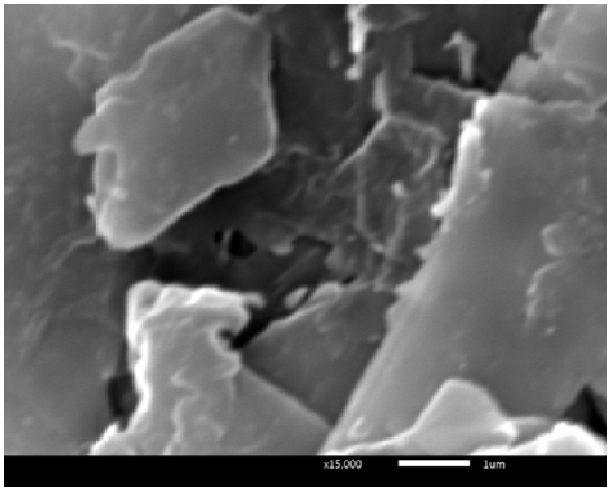

Figure 3a. SEM snapshot of powder
20 kV, PC 60, WD 11, M 15000

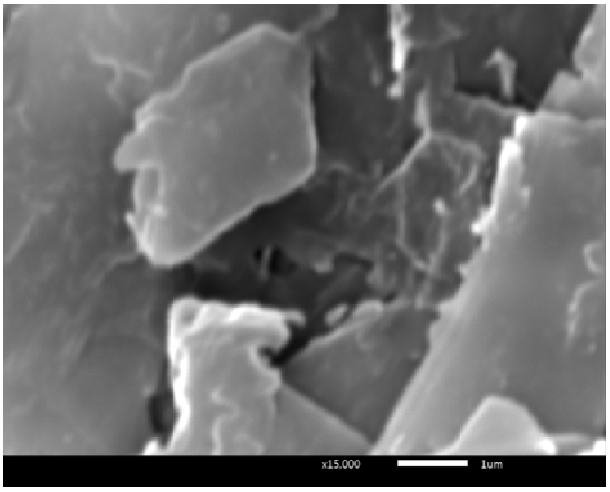

Figure 3b. SEM snapshot of powder
20 kV, PC 70, WD 11, M 15000

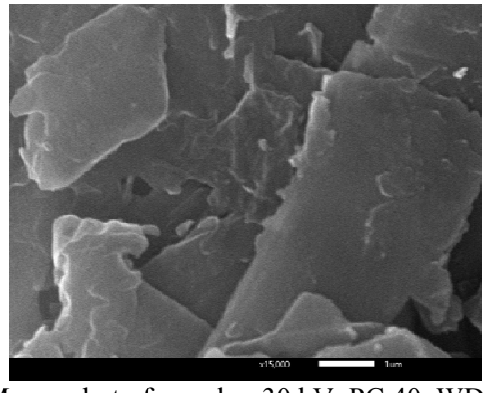

Figure 4. SEM snapshot of powder, 30 kV, PC 40, WD 7.6, M 15000

The difference between structures recorded by SED and BED type are given in Figure 5.

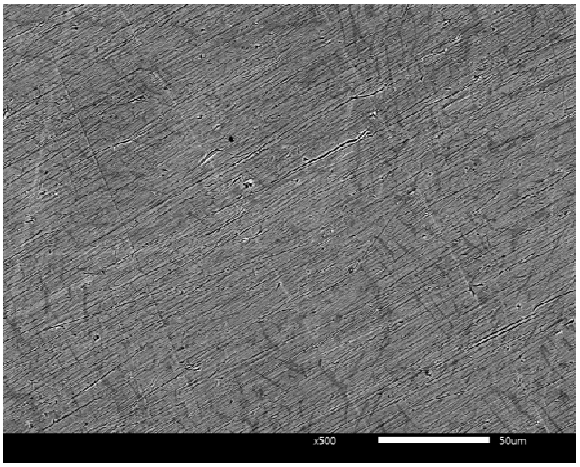

Figure 5a. SEM snapshot (BED mode) of shape memory alloy 20 kV, PC 67.2, WD 10, M 500

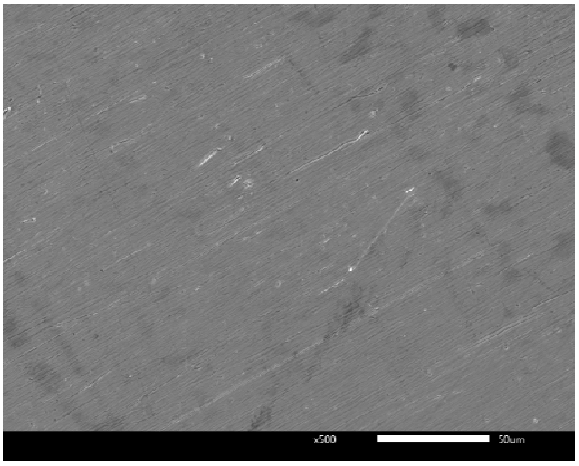

Figure 5b. SEM snapshot (SED mode) of shape memory alloy 20 kV, PC 67.2, WD 10, M 500

4. CONCLUSION

The factors which have influence on the SEM-EDS quality and reliability are numerous. In this paper, just a few examples of those factors were presented. Voltage, probe current, working distance are settings which are related with any SEM-EDS and the quality of results depends only of operator itself.

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