Application Brief
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Analysis of 15 nm Iron Nanoparticles in Organic Solvents by spICP-MS

Using the exceptional sensitivity and low background of the Agilent 8900 ICP-QQQ

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Monitor metallic nanoparticles in process chemicals

In semiconductor device manufacturing, even small amounts of impurities present in processing reagents can affect product yield and reliability. There is growing awareness that metallic nanoparticles (NPs), especially Fe NPs, can lead to the occurrence of defects on the surface of wafers. Single particle ICP-MS (spICP-MS) is a powerful tool that is used increasingly to characterize the NP content of various types of samples, including semiconductor process chemicals.

Measurement of 15 nm Fe NPs using spICP-MS

The Agilent 8900 ICP-QQQ (semiconductor configuration) was fitted with a quartz torch with a 1.5 mm i.d. injector. Interferences on ^{56}Fe from ArO and C_2O_2 were resolved using oxygen as the cell gas. The Agilent SPS 4 autosampler was used, so large bottles (100 to 500 mL) could be used for long-term stability tests. The samples were self-aspirated using an Agilent PFA nebulizer with SPS 4 probe kit (G3139-68000). Data analysis was performed using the optional Single Nanoparticle Application module of Agilent ICP-MS MassHunter software.

Table 1. 8900 ICP-QQQ operating parameters used for spICP-MS method.

Parameter	Setting	Parameter	Setting
RF Power (W)	1200	Energy Discrimination (V)	–8.0
Sampling Depth (mm)	16	Cell gas (O_2) Flow (mL/min)	0.38 (25%)
Neb Gas Flow (L/min)	0.75	Dwell Time (us)	100
Makeup Gas Flow (L/min)	0.5	Scan Mode	Single quad mode
*Option Gas (O_2) Flow (L/min)	0.12 (12%)	Mass Monitored	56 (Fe)
Spray Chamber Temp (deg)	–2	Data Acquisition Time (s)	60
Axial Acceleration (V)	2		

**Direct injection of organic solvents was possible by adding oxygen (50% balanced with Ar) to prevent the deposition of carbon on the cones.*

The spICP-MS method was used to measure isopropyl alcohol (IPA), propylene glycol methyl ether acetate (PGMEA), and propylene glycol monomethyl ether (PGME) spiked with 15 nm Fe_2O_3 NPs (Sigma Aldrich). Figure 1 shows the signal distribution (upper) and size distribution plots (lower) for Fe NPs in each of the samples. The NP signals were clearly separated from the background signals. Also, the mean measured particle size was around 15 nm in all spiked samples, which is consistent with the nominal Fe NP diameter of 15 nm.

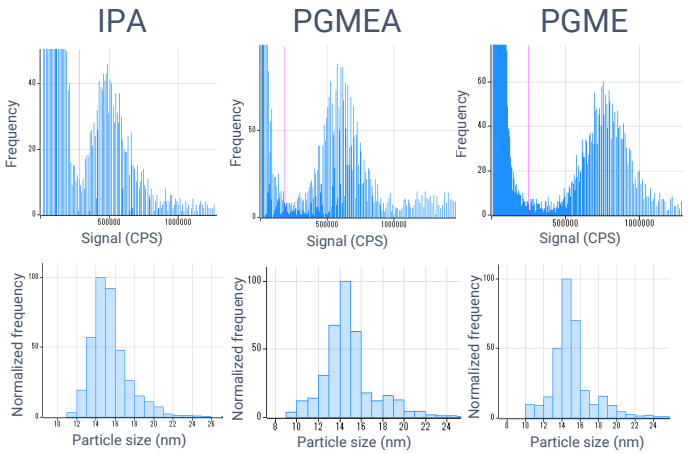

Figure 1. Signal distribution (upper) and size distribution (lower) of 15 nm Fe NPs in solution of IPA, PGMEA, and PGME.

Size ratio of 15 and 30 nm particles

Table 2 shows the average signal intensities from one particle with a 30 nm diameter and one particle with a 15 nm diameter, and the ratio of these intensities (30 nm/15 nm). As the signal intensity is proportional to the cube of the diameter of the particle, the ratio of signal intensities from the 15 and 30 nm NPs should be 8. The measured ratio was 8.44, which is acceptable, considering the accuracy of the nominal diameters.

Table 2. Average particle signal intensities and ratio of the intensities.

Average signal intensity from one particle (cps)		Ratio (30 nm/15 nm)
30 nm	15 nm	
5,216,482	617,736	8.44

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Long-term stability tests

Figure 2 shows the stability of detected number (representing particle number concentration) and particle size for 15 nm Fe NPs in two of the solvents over 10 hours. Both the detected particle number and the size were constant over 10 hours, as indicated by the %RSDs.

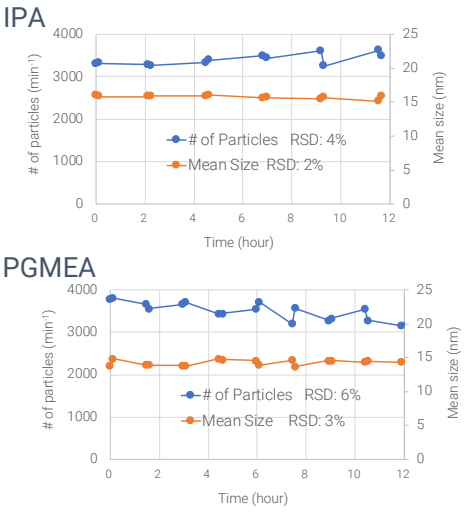

Figure 2. Long-term stability of 15 nm Fe NPs in IPA and PGMEA over 10 hours.

Figure 3 shows the size distribution profiles of 30 nm Fe NPs in IPA on the day of sample preparation and six months later. After six months, the signals from 30 nm Fe NPs were clearly observed and the shape of the size distribution is almost the same as the one in the fresh solution. The results show that Fe NPs are stable in IPA for a long period, suggesting that they don't dissolve into or precipitate out of the solvent.

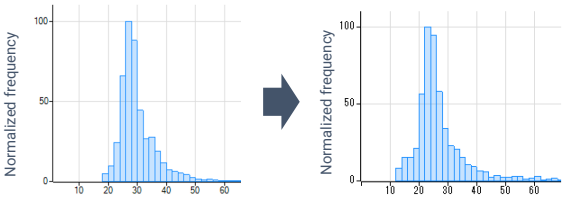

Figure 3. Size distribution of 30 nm Fe NPs in IPA on the day of preparation (left) and six months later (right).

Single particles in high purity solvents

The 8900 spICP-MS method satisfies the emerging needs of the semiconductor industry to monitor low concentrations of small sized particles in high purity solvents.

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