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МЕТАЛЛИЧЕСКИЕ ПОВЕРХНОСТИ И ПЛЁНКИ

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New Method and Tool for TEM Samples Preparation

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A new tool for TEM sample preparation, which allows preparing a thin lamella with thickness less than 20 nm surrounded by and embedded in bulk material, is presented. The main advantages of this system are low ion milling induced damage (less than 2 nm in depth), low process time (1–2 hours), in situ sample monitoring during ion milling (topography and sample thickness), and large treated area (5–30 μm along the area of interest). Comparison of few kinds of working substance of ion sources as well as schemes or drawings of key components of the tool are presented.

Представлено нову методику і технологію виготовлення зразків для ПЕМ, яка уможливлює підготувати зразок завтовшки до 20 нм, оточений об'ємним матеріалом і втілений у нього. Головна перевага системи полягає в малій товщині шару пошкоджень, спричинених йонним обробленням (менше 2 нм), в малому часі оброблення (1–2 години), в моніторинґу зразка під час йонного фрезерування (топографія і товщина зразка) і у великій площі, що обробляється (5–30 мкм вздовж області, яка являє інтерес). Виконано порівняння декількох типів робочої речовини для йонних джерел, наведено схеми або креслення ключових вузлів пристрою.

Представлена новая методика и технология приготовления образцов для ПЭМ, позволяющая подготовить образец толщиной до 20 нм, окружённый объёмным материалом и вмурованный в него. Главным преимуществом системы является малая толщина слоя повреждений, индуцированных ионной обработкой (меньше 2 нм), малое время обработки (1–2 часа), мониторинг образца во время ионного фрезерования (топография и толщина образца) и большая обрабатываемая площадь (5–30 мкм вдоль интересующей области). Проведено сравнение нескольких рабочих веществ для ионных источников, приведены схемы или чертежи ключевых

узлов устройства.

Key words: ion milling, thin lamella, radiation damage.

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1. INTRODUCTION

Preparing of thin coplanar high quality samples for TEM with minimal preparation induced damage is a key problem on transmitted electron microscopy as a method of analysis in electronics. Currently, the CMOS (Complementary Metal–Oxide–Semiconductor technology) manufacturing is based on the 32 nm technological process. Now, the 20 nm thick samples seem reasonable and the damage induced by preparation procedure of a work piece must not exceed 1–2 nm.

The easiest way is to etch a work piece by a beam of accelerated ions. Etching by Ar^+ ion beams (PIPS[1]) or focused Ga^+ ion beams (FIB[2]) are most prevalent. These technologies failed to solve this issue [1]. In 2004, the necessity to develop a new system for TEM samples preparation was realized. Listed below are the following conditions to be met:

– ion etching process must occur when using stationary sample with the ability to control the quality and the thickness of the specimen in real time, using scanning electron microscope with resolution of 50–100 Å;

– at least 5-degrees of freedom manipulator is required for specimen handling;

– availability of ion source with resources up to 2000 hours (up to 6 months in operation) and the possibility to focus the ion beam to several tens of micrometres in diameter;

– ion beam management should be purely electrostatic with the possibility to alter its focus, angle of incidence and energy of ions in a wide range;

– sample preparation procedure should be fully automated and process time must not exceed of 2 hours.

In a given article, the conditions mentioned above and the intense and innovative efforts responsible for the success of the new method will be discussed.

2. INSTRUMENTATION

Commercial designation of the system is Xact[3]. This system utilizes AIM™ (Adaptive Ion Milling) technology.

Xact equipped (Fig. 1) with commercial scanning electron micro-

[1] http://www.gatan.com/specimenprep/691_pips.php
[2] http://www.fei.com/products/focused-ion-beams/
[3] http://www.camtek.co.il/php/index.php?option=com_content&task=view&id=313&Itemid=249

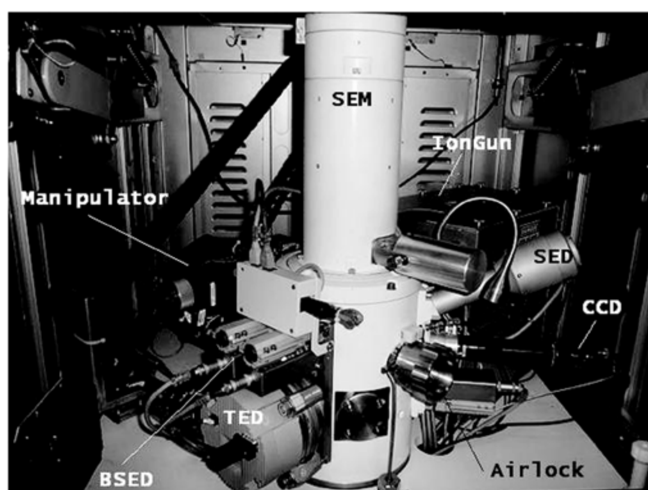

Fig. 1. Xact general view.

scope. The resolution of the microscope is 6 nm on the distance of 18 mm. The SEM image is generating via detectors of secondary electrons (SED), back scattered electrons (BSED) and primary transmitted electrons (TED). The BSED is a multidetector (A, B) that allows receiving two types of images: topographic (difference of A and B signals) and composition (sum of A and B signals). The TED is composed of three concentric detectors allowing receiving of bright field and dark field images and measuring (control) thickness of a work piece beginning from 6 nm (Fig. 2).

Vacuum in the system is supported by the turbomolecular pump with 400 l/s (N_2) efficiency at the level of $(2.53–3)\cdot 10^{-6}$ torr. Vacuum in the area of tungsten filament of the microscope is supported by the ion pump at the level of $2\cdot 10^{-7}$ torr.

The process of mounting the sample on the manipulator and loading it into the chamber takes approximately 1 minute. 5-degrees of freedom manipulator (X, Y, Z, R, T, where R—rotation around X-axis, T—rotation around Z-axis) is designed for accurate positioning of the sample for milling and observation.

The FIB has been developed utilizing LMIS (Liquid Metal Iron Source)—high brightness ion source. The FIB small beam size is achieved due to low ion current and high accelerating voltage (30–50 kV). Low ion current does not allow removing bulk of sample's material (to open wide area of interest). Increased ion energy leads to the development of extensive damaged layer on sample's surface. The ion source used in the system has several important characteristics: high brightness ($\cong 300$ $A\cdot m^{-2}\cdot sr^{-1}\cdot V^{-1}$), high ion current (up to 30 μA) and low vacuum load (working pressure is $\cong 3\cdot 10^{-6}$ torr). The only adjustable parameter of the

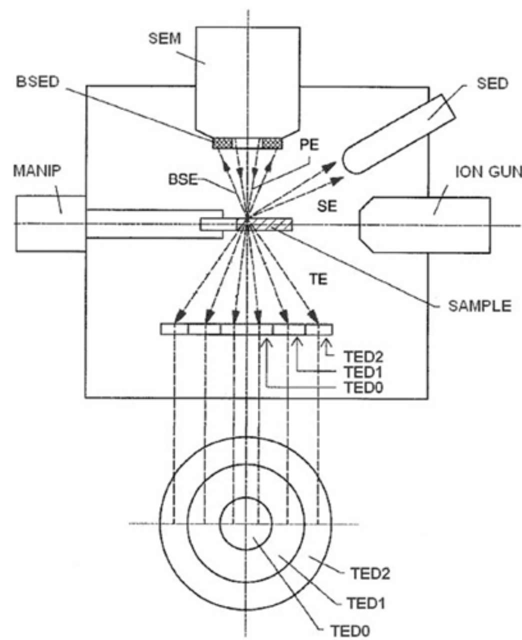

Fig. 2. Xact scheme.

ion source is discharge voltage, which is stabilized by automated managing of the leak valve flow rate. Thermomechanical leak valve appeared to be the easiest and most reliable. The flow rate of the leak valve was managed by controlled thermal expansion (CTE) of high-CTE body relative to invar (CTE = 0.6 μm/m·°C) closing pin. The time constant of the feedback circuit (few seconds) correlated well with the working gas pressure variation time in the discharge area. The leak valve and ion-beam-accelerating electrode are of the same potential (up to 10 kV). Therefore, to prevent breakouts, HV isolation is provided from the high-pressure side (usually 110 kPa) of the working gas line (Fig. 3).

An ion source of this kind allows the generation of an ion current of 30 μA at accelerating voltage of 8 kV and 0.5 μA at accelerating voltage

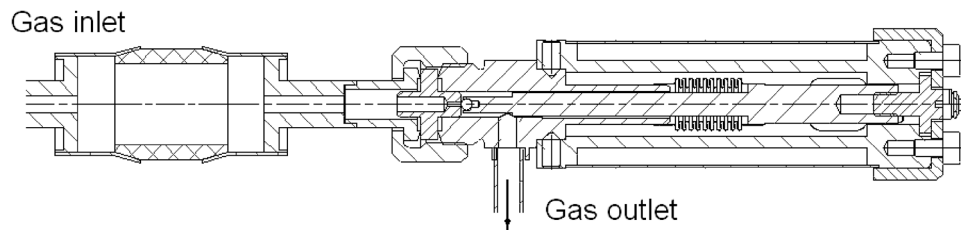

Fig. 3. Leak valve.

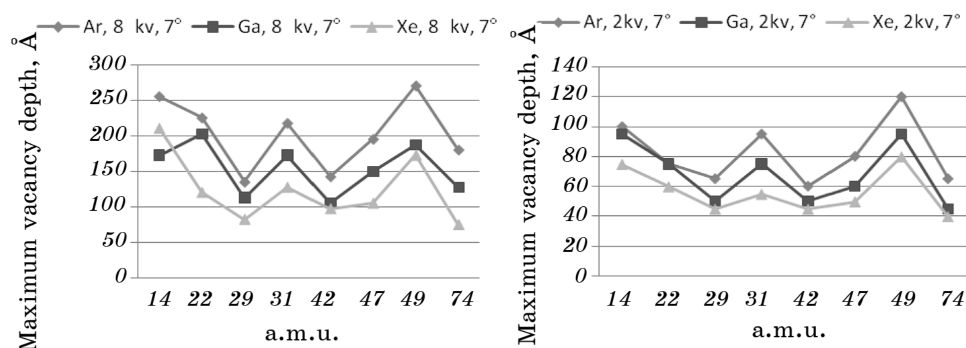

Fig. 4. Maximum vacancy depth for different kinds of ions.

of 1 kV. The generating and accelerating of ions are separated. This significantly simplifies the adjustment and managing of the ion source, thereby allowing working in a wide range of energies. Ion source time to service depends on the time needed for sidewall of hollow cathode to be sputtered through and it is more than 6 months of continuous work.

3. WORKING SUBSTANCE AND OPTICAL SYSTEM

Ions of Ar^+ or Ga^+ are usually used for etching, particularly, in TEM sample preparation. Analysis of amorphous and damaged layers caused by ion etching revealed reduction of ion beam induced damage while increasing the ion's mass. Simulation of this effect was made utilizing TRIM (Transport of Ions in Matter) SW for Ga^+, Ar^+, and Xe^+ ions (Fig. 4, Tables 1 and 2). Range — depth the primary ion penetrates to; Yield

TABLE 1.

		Ar, 8 kV, 7°			Ga, 8 kV, 7°			Xe, 8 kV, 7°		
	a.m.u.	Range	Yield	Vac. depth	Range	Yield	Vac. depth	Range	Yield	Vac. depth
Si	14	50	12.20	255	35	16.10	173	31	20.20	210
Ti	22	42	7.60	225	30	10.00	203	24	12.30	120
Cu	29	29	17.30	135	19	21.20	113	15	25.60	83
Ga	31	49	13.90	218	30	16.20	173	23	20.30	128
Mo	42	33	7.30	143	24	9.30	105	17	11.10	98
Ag	47	41	14.00	195	26	18.20	150	17	20.70	105
In	49	62	13.70	270	40	17.30	188	26	21.10	173
W	74	39	5.80	180	25	7.80	128	15	9.40	75

TABLE 2.

		Ar, 2 kV, 7°			Ga, 2 kV, 7°			Xe, 2 kV, 7°		
	a.m.u.	Range	Yield	Vac. depth	Range	Yield	Vac. depth	Range	Yield	Vac. depth
Si	14	21	5.80	100	18	7.10	95	17	8.60	75
Ti	22	18	3.70	75	15	4.50	75	12	5.40	60
Cu	29	12	7.30	65	9	8.20	50	9	9.30	45
Ga	31	20	6.30	95	15	7.30	75	13	8.50	55
Mo	42	16	3.30	60	11	3.80	50	9	4.20	45
Ag	47	18	6.40	80	13	7.20	60	10	7.90	50
In	49	27	6.60	120	19	7.40	95	14	8.40	80
W	74	17	2.60	65	12	3.20	45	9	3.50	40

— number of sputtered atoms per incident ion; Vac. depth — depth of structure distortions.

It appears that usage of Xe as a working gas reduces the depth of damaged layer by 20–40%. In addition, noble gases do not interact chemically with target material. Another advantage is the low potential of ionization of Xe (12.13 eV) relative to Ar (15.8 eV) which ensures a more stable ion source discharge.

The most innovative Xact component solved the initial issue—the multideflecting system for the ion beam. The final scheme of ion optics is shown in Fig. 5.

The ion beam extracted from anode by the extractor (1) is focused by first condenser lens (2) to 1.5–2 mm dia. Beam tails are cut by the diaphragm (3). The position of the beam is corrected according to system's axis by first deflecting unit (4). Then second long-focus low spherical

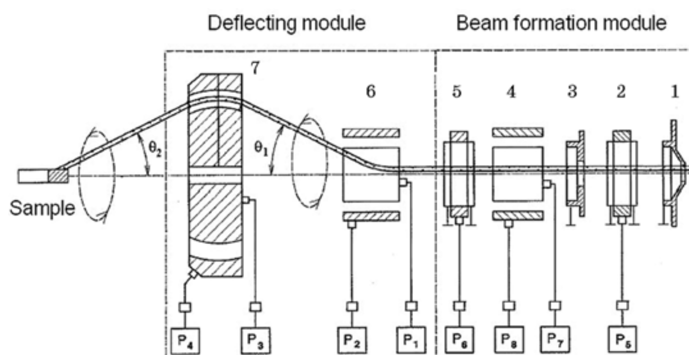

Fig. 5. Scheme of ion optics of Xact.

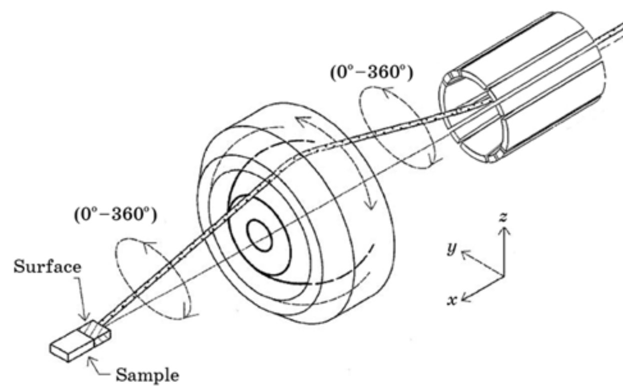

Fig. 6. Spherical deflectron.

aberrations lens (5) forms a paraxial slightly convergent I-beam. Second deflecting unit (6) is capable of deflecting the I-beam 7 degrees from system's axis without distortions. Computer simulation (Simion) and experiment have confirmed that conical dodecapole (12 poles deflector) [2] can handle this. Deflected beam reaches the key unit of the system—spherical deflectron (7).

It is a slice of spherical condenser (Figs. 6, 7) where θ_2 is the maxi-

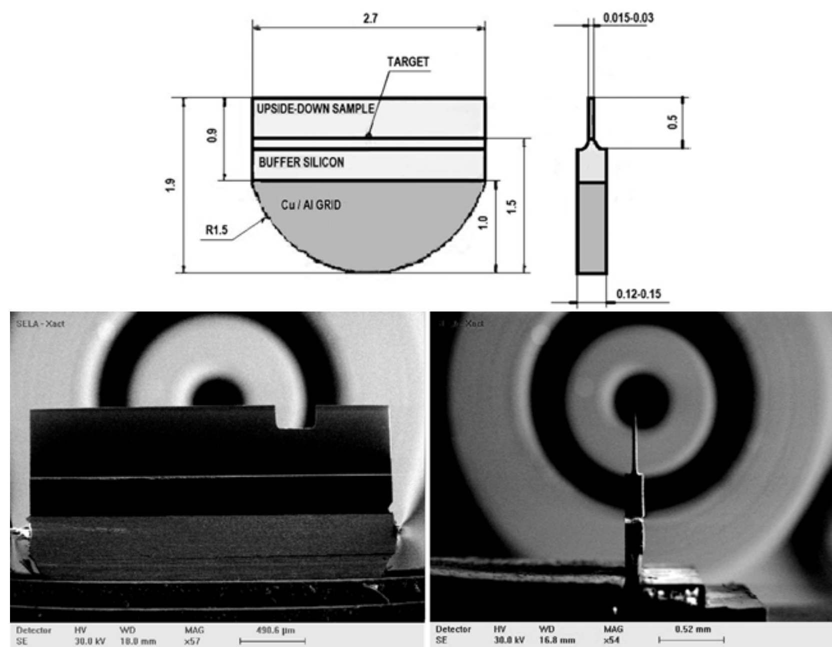

Fig. 7. Initial sample.

mum angle of incidence into horizontal sample. It is known that spherical condenser is the deflecting, stigmating and focusing element.

The point object, centre of the sphere, and point image of the object are located on the same line. This characteristic made it possible to position all of the ion-optical elements and an area of interest of the sample into the same axis. The area of interest of the sample coincide with the intersection of ion gun and SEM axes, so it is possible to examine the sample during preparation.

Actual dimensions of spherical deflectron are defined by the working distance of SEM and maximum angle of incidence of ion beam into the sample. With a maximum angle of incidence of 60 degrees (from the normal to surface) that corresponds to the angle close to maximum sputtering rate [3], the distance between the centre of sphere and electron beam equals to diameter of sphere. Angle of incidence of the ion beam to the sample varies by polar angle variation according to the formula:

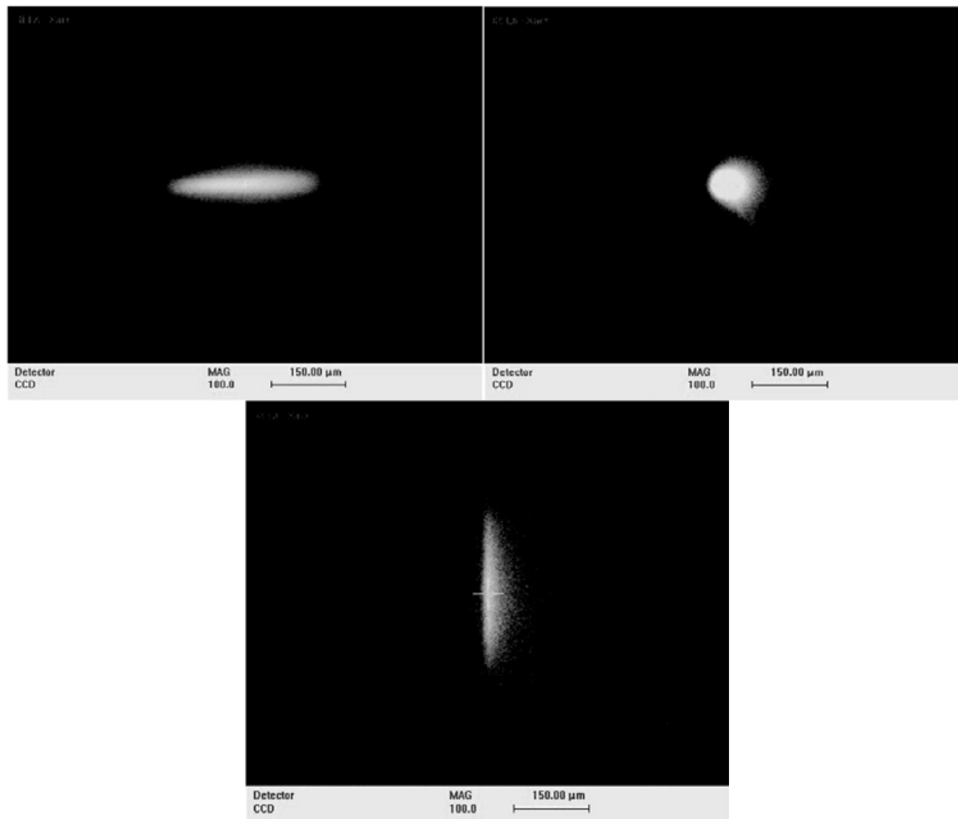

Fig. 8. Ion beam visualization.

TABLE 3.

Automatic sub-process	Incident angle of ion beam, degrees	Thickness of a sample after each sub-process, nm
Trenching milling, 8 kV	30	3500
Glancing milling, 8 kV	4	500
Glancing milling, 4 kV	4	350
Glancing milling, 3 kV	4–5	200
Glancing milling, 2 kV	5–6	100
Glancing milling, 1.5 kV	5–7	50 and below

$$2\sin(\text{incidence}) = \sin(\text{polar angle}),$$

i.e. a thin static sample could be treated from both sides with an ion beam with angles of 0–30 degrees (Fig. 6).

The main purpose of the system is the preparation of thin lamellas of the IC cross-section. Prior to Xact treatment, samples are cryogen cut from a wafer by EM3[4] [1]. Configuration of a sample is shown on the Fig. 7. The target area is masked by the substrate silicon, which in any case (utilizing of any sample preparation tool) could be a contaminant.

After EM3 cutting, the sample is located in a relatively big clamp, so, it is safe and easy to manipulate. Further work is a piece thinning resulting ideally—in thin lamella (20–50 nm) supported by bulk silicon, usually—low angle wedge.

Xact has an ion beam visualization system. Possible beam shapes are shown on Fig. 8: horizontal ellipsis, circle, and vertical ellipsis.

The Table 3 lists the typical sequence of TEM (HRTEM) sample preparation.

Xe^+ ion beam induced damage was measured for the Xact and compared to damage caused by other systems utilizing Ar^+ and Ga^+ [1]. Dependence is as follows: ≈ 1nm of damaged layer for 1 keV of ion energy (for 3–7 degrees incidence). For the first time, direct measurement of amorphous layer thickness of Si caused by Xe^+ ions bombardment was performed [4]. It was shown that as low as 1–1.8 nm of amorphous layer thickness is feasible. In Figure 9, HRTEM image shows the depth of damaged layer on crystalline silicon.

Preparation of the sample is described in detail in the article [4]. However, the protective Cu layer has not been applied. It had no influence on the result.

The Table 4 shows the comparison of popular methods of TEM sample preparation.

[4] http://www.camtek.co.il/php/index.php?option=com_content&task=view&id=314&Itemid=250

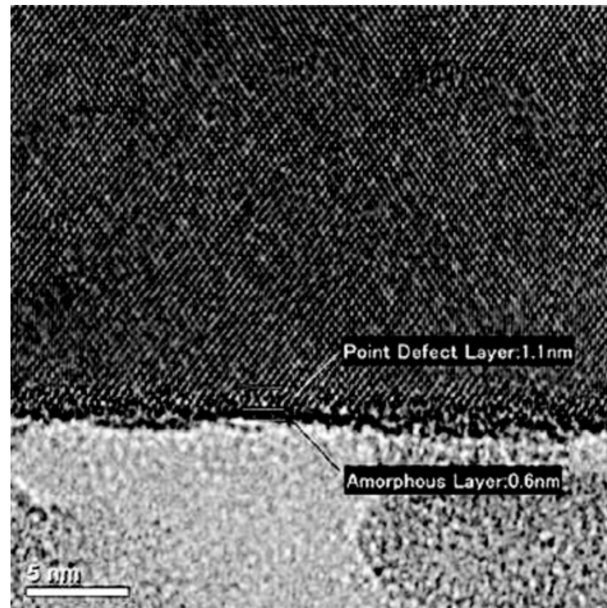

Fig. 9. Cross-section of crystal silicon exposed by 7°, 1.5 keV Xe^{+} beam. Top-down: c-Si, damaged Si layer, glue (bright).

TABLE 4.

	FIB	BIB	AIM
Pre-processing	yes/no*	required	required
Sample handling	difficult	difficult	easy
Process control	good	poor	good
Target localization	good	poor	good
Auto endpoint detection	no	no	yes
In situ imaging	yes	no	yes
Treated area	small	large	large
Minimum accelerating voltage, V	500	100	500
Sample quality	fair	good	very good
Ga/Ar/Xe contamination	heavy Ga	low Ar	low Xe
Artefacts removing	Ar milling**	not required	not required
Time consumption	1–4 hours	not relevant***	1–2 hours

* depends on FIB process; ** no further milling possible for some processes; *** manual handling dependent.

4. CONCLUSION

The new method and tool for TEM samples preparation has been developed and successfully introduced in laboratory practice for examination of electronic components with the lowest level of radiation damage currently achieved at global level (1 nm). Further reduction of this important value may be obtained by lowering the energy of bombarding ions but a source with a higher brightness is needed for this purpose.

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