Effective removal of field-emitting sites from metallic surfaces by dry ice cleaning

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(Received 27 February 2007; accepted 6 July 2007; published online 21 August 2007)

Systematic results of the field emission properties of polycrystalline copper and niobium and single-crystal Nb are reported. Dry ice cleaning (DIC) is found to suppress enhanced field emission from metallic surfaces. The cleaning effect on the emitting sites was investigated by means of field emission scanning microscopy up to fields of 250 MV/m and high-resolution scanning electron microscopy with energy dispersive x-ray analysis. The number density of emitters at given fields was drastically reduced by dry ice cleaning. Current-voltage measurements and derived Fowler-Nordheim parameters are partially discussed with respect to the morphology and impurity content of localized emitters. No emission from grain boundaries on large-grain Nb samples was observed. The microscopy results prove the effective removal of field-emitting particulates down to 400 nm as well as the partial smoothing of surface protrusions by DIC. © 2007 American Institute of Physics. [DOI: 10.1063/1.2772505]

I. INTRODUCTION

Development of effective cleaning techniques for the removal of small particulates from metal surfaces is a primary need in high-voltage vacuum devices.¹ Micron size surface irregularities can seriously affect the performance of high field accelerator cavities² by creating field emission (FE) sites,³ from where electrons are emitted at much lower electric fields than predicted by Fowler-Nordheim (FN) theory.⁴ This enhanced FE is mainly caused by particulates⁵ and surface protrusions⁶ due to local field enhancement. Such emitters originate as residues of chemical surface preparation and cleaning techniques, or result from insufficient cleanroom conditions and mishandling of the surface.^{7–10}

The removal of particulate emitters is rather difficult because these usually stick to the solid surface due to adhesion. The interaction between particulates and substrate includes van der Waals, electrostatic, and capillary forces.¹¹ All these adhesion forces F are found to be proportional to the particulate diameter d . Accordingly, a particulate of mass $m \sim d^3$ will require an acceleration $a = F/m \sim 1/d^2$, which becomes quadratically larger with decreasing size. Additionally, the number density of ambient particulates usually increases with decreasing size. Therefore, some small particulates usually remain on the surface after conventional cleaning techniques used in accelerator technology such as high-pressure rinsing (HPR) with ultrapure water.¹²

Dry ice cleaning (DIC) is a powerful technique which uses a high-pressure jet of pure carbon dioxide snow to loosen and remove different types of particulate contaminations from the surface by its unique combination of thermal, mechanical, and chemical effects.^{13–16} The rapid cooling at

the point of hitting brittles the particulates, thus weakening the adhesion for their efficient removal by the impact of snow particles. Unlike HPR, this technique significantly removes hydrocarbon contaminations from the cleaned surfaces,¹³ since carbon dioxide is a good solvent for nonpolar chemicals. The possibility of a dry *in situ* cleaning of the cavities, and already proven faster pump-down rates with DIC,¹⁷ are surely advantageous for accelerator technology.

Nowadays copper and niobium sheet metals are used for the fabrication of cavities for accelerators. Dark currents caused by enhanced FE limit the accelerating field gradients in normal conducting rf guns¹⁸ and superconducting Nb cavities.¹⁹ Therefore, systematic investigations of the dc field emission properties of relevant metallic samples has proven to be helpful for the optimization of surface preparation techniques. In order to understand the origin of the emitters, field emission scanning microscopy (FESM),^{6,20} combined with high-resolution secondary electron microscopy (SEM) and energy dispersive x-ray analysis (EDX), is required.¹⁰

Here, we report on the effect of DIC on Cu and Nb surfaces by (i) comparing the number density of FE sites on particular sample areas scanned in FESM before and after DIC; (ii) determining the FE properties of localized emitters by current versus voltage (I – V) measurements and FN analysis; and (iii) investigating their morphology and composition by SEM and EDX. This comprehensive study will prove that DIC reduces FE efficiently by removal of micron and submicron particulates as well as by smoothing of surface protrusions.

II. EXPERIMENTAL TECHNIQUES

We have tested four copper (7 mm squares) and six niobium (28 mm diameter) samples, of which two were always fabricated with the same techniques for reproducibility

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FIG. 1. (Color online) High-pressure dry-ice jet emerging from DIC nozzle.

checks. Two polycrystalline Cu samples were cut from a regularly turned cavity cell and the other two from a single-point diamond-turned cavity cell. Two polycrystalline Nb samples (PolyNb) were machined from high-purity niobium with a residual resistance ratio RRR of about 300 and electropolished up to $140\text{ }\mu\text{m}$ using $\text{H}_2\text{SO}_4\text{:HF}$ in 9:1 volume ratio, resulting in a surface roughness of less than $0.5\text{ }\mu\text{m}$. Two large-grain ($\sim\text{cm}$) and two single-crystal Nb samples (CryNb) of $\text{RRR} \sim 340$ were chemically etched by buffered chemical polishing ($\text{HF:HNO}_3\text{:H}_3\text{PO}_4$ in volume ratio 1:1:2) to remove the damage layer up to $30\text{ }\mu\text{m}$, resulting in a mirrorlike surface with surface roughness of less than $0.1\text{ }\mu\text{m}$. Finally, all samples were extensively rinsed with ultrapure water and HPR in the case of Nb. All the samples have been especially marked during fabrication to adjust the sample position in different experimental setups. For the sample transfer between the laboratories, an approved protection cap system²¹ was used and opened under cleanroom or high-vacuum conditions only.

A modified DIC of the samples was performed in a class 10 cleanroom. Detailed description of the DIC experimental setup and the cleaning procedure has been described elsewhere.^{22–24} Carbon dioxide of purity grade 4.5 ($\text{N}_2 + \text{O}_2 < 45\text{ vol ppm.}$, $\text{H}_2\text{O} < 5\text{ vol ppm.}$, $\text{C}_n\text{H}_m < 1\text{ vol ppm.}$) and pure nitrogen (both particle filtered to $< 0.05\text{ }\mu\text{m}$) are supplied up to pressures of 50 and 12 bar, respectively. Figure 1 shows the high-pressure DIC jet coming out of a dedicated nozzle system developed at Fraunhofer Institut Produktionstechnik und Automatisierung (Stuttgart, Germany) for DESY for the cleaning of cavities. The CO_2 snow jet is surrounded by supersonic nitrogen,²² which provides additional acceleration and focusing of the jet as well as a partial pre-

FIG. 2. (Color online) Inner view of FESM, showing a mirrorlike Nb sample of 28 mm diameter below a tungsten tip anode ($\sim 1\text{ }\mu\text{m}$ apex radius).

FIG. 3. (Color online) FE maps of Cu, performed with a conical anode ($100\text{ }\mu\text{m}$) over the same ($5 \times 5\text{ mm}^2$) area before (left) and after (right) DIC at 120 and 150 MV/m, respectively.

vention of moisture condensation at the cleaned surface. The temperature of liquid CO_2 is maintained between $-18\text{ }^\circ\text{C}$ and $-10\text{ }^\circ\text{C}$ at the dedicated cooler/purifier unit,¹⁶ which is connected to the nozzle by a pipe of 3 m length. This configuration keeps the fraction of snow in the jet on the order of 45%.²² An effective gas extraction system and a controlled heating system avoid recontamination and humidity condensation at the sample surface, respectively. The flat samples were cleaned by moving the DIC nozzle linearly back and forth with a constant speed. The cleaning duration of the large samples (28 mm diameter) was usually about 5 min, which includes the intermediate time for rotating the sample six times manually by 30° .

FESM measurements have been made in two steps: (i) scans over a selected sample area with a fixed anode at a maximum field given by a high applied voltage V regulated for a constant current I (2 nA) within a few milliseconds, resulting in maps of the found emitters at correspondingly reduced field level; and (ii) local characterization of the interesting emitters observed in the scans. The scans were performed at field levels varying as 60, 90, 120, 150, 200, and 250 MV/m for different samples by using a 5 kV power supply and adjusting the gaps between sample and anode down to $20\text{ }\mu\text{m}$. For the scans conical anodes of diameter 300 or $100\text{ }\mu\text{m}$ were used depending on the area and required resolution. Local measurements were mostly performed with tip anodes of diameters 30, 10, or smaller down to $1\text{ }\mu\text{m}$ to resolve small emitting sites. Figure 2 shows a tip anode of apex radius $1\text{ }\mu\text{m}$ placed just above an emitting site on a large-grain Nb sample. The mirrorlike sample surface is helpful to control the gap within an accuracy of $\pm 5\text{ }\mu\text{m}$ by observing the anode image with a long-distance microscope and charge-coupled device (CCD) video camera. The real electrode distance z is derived prior to any local $I - V$ curve of an emitter by extrapolation of the measured $V(z)$ plot.²⁰ The local onset field for a current of 2 nA, $E_{\text{on}} = V/(z\alpha)$, has been corrected for the calculated tip shape factor α .

The relocation of the emitters found with FESM in an *ex situ* SEM has been ensured within an accuracy of $\pm 500\text{ }\mu\text{m}$ by using the marks on the samples. The high-

FIG. 4. (Color online) FE maps of polycrystalline Nb, performed with a conical anode ($100\ \mu\text{m}$) over the same (7.5×7.5) mm^2 area before (left) and after (right) DIC at 120 MV/m.

resolution SEM images and EDX measurements (for elements with $Z > 10$) were made by means of a Philips XL-30 with LaB_6 cathode.

III. RESULTS AND DISCUSSION

A. Statistical reduction of FE by DIC

FE scans of two Cu samples (one of each type) performed with $100\ \mu\text{m}$ conical anode over the area of (5×5) mm^2 showed no emission at 60 MV/m before DIC. For the diamond-turned sample, 10 emitters have been found at 120 MV/m as shown in Fig. 3(a), which reduced to 1 after DIC. This improved surface gives 6 emitters at 150 MV/m [Fig. 3(b)]. The regularly turned Cu sample gave 8 emitters at 90 MV/m, which improved after DIC to 0 (3) emitters at 90 (120) MV/m, i.e., very comparable results. Therefore, the remaining Cu samples were directly dry ice cleaned. All four DIC Cu samples showed no FE up to 90 MV/m, and the best one up to 120 MV/m. No significant difference was found between the regularly and diamond-turned Cu samples.

The PolyNb samples were first scanned with $300\ \mu\text{m}$ anode over the area of (12×12) mm^2 at low fields (40, 60, 90 MV/m) and then with $100\ \mu\text{m}$ anode over (7.5×7.5) mm^2 area at 120 MV/m. Both samples showed before DIC first emission of (1, 2) emitters at 60 MV/m, which disappeared

FIG. 5. (Color online) FE maps of single-crystalline Nb, performed with a conical anode ($100\ \mu\text{m}$) over the same (5×5) mm^2 area before (left) and after (right) DIC at 200 and 250 MV/m, respectively.

FIG. 6. (Color online) Emitter number density vs applied electric field for showing the improvement by DIC for the different materials. (Dark symbols (red online): before DIC, and grey symbols (green online): after DIC).

after DIC and improved to (2, 0) emitters at 90 MV/m and (2, 3) at 120 MV/m. The great improvement after DIC for one of the samples is shown in Fig. 4. Only one emitter survived but has been weakened by DIC, as seen from the field scales of the maps. For a more detailed interpretation, strong emitters were investigated locally in FESM and SEM and will be discussed in Secs. III B and III C.

FE maps of CryNb samples have shown very good results even before DIC: for large-grain Nb (0, 2) at 120 MV/m, (5, 3) at 150 MV/m, and (10, 12) at 200 MV/m, and for single-crystal Nb no emission up to 120 MV/m, (2, 1) emitters at 150 MV/m, and (5, 9) at 200 MV/m. It is most interesting that DIC further improves such high-quality surfaces. Three samples, one large-grain Nb and two single crystal, were dry ice cleaned and showed no FE up to 150 MV/m, but (2, 0, 1) emitters at 200 and 250 MV/m. FE maps of one sample at the maximum fields before and after DIC are given in Fig. 5. The main observations are (i) removal of all old emitters after DIC, showing the effectiveness of this cleaning technique; and (ii) existence of one new emitter at 250 MV/m (which occurred already at 200 MV/m).

A statistical overview of the number density of emitters N for the varying electric field E is presented in Fig. 6. In order to reduce the statistical error and to simplify the $N(E)$ plot, we summed up all the results for a particular kind of sample, i.e., the corresponding areas and number of emitters were added at the scanned field levels. Thus, only six lines for the three different kinds of samples before and after DIC summarize all the scan results. From the statistical plot, the beneficial effect of DIC on these metallic surfaces is obvious: (i) shift of $N(E)$ curves to right, i.e., onset of FE observed at higher fields; and (ii) $N(E)$ curves with much reduced slope, i.e., huge reduction of the number density of field emitters at a given field level. Within statistical errors, for Cu and PolyNb no FE was observed up to 60 MV/m before and 90 MV/m after DIC, and for CryNb up to 150 and 200 MV/m, respectively. It is remarkable that one of the DIC single-crystal samples did not show any FE up to 250 MV/m.

B. FN analysis and stability of emitters

The majority of the localized emitters on all scanned samples were analyzed with FESM. The current-voltage

FIG. 7. I – V curve for a typical emitter on dry ice cleaned Cu sample, with β , S values derived for $\Phi=4.4$ eV from its FN fit (solid line).

characteristics of the emitting sites were found to obey the modified FN law.³ Accordingly, the linear fitting of $\ln(I/E^2)$ vs $1/E$ plot gives the field enhancement factor β for a given work function Φ from its slope and the effective emission area S from its intercept. Figure 7 shows an I – V curve for a typical emitter on DIC Cu surface, resulting in a characteristic β value of 45 and S value of $0.005 \mu\text{m}^2$. These values seem to be reasonable for a particulate or protrusion of size $h(\sim\mu\text{m})$ and sharp edge radius $r(\sim 10 \text{ nm})$, since $\beta=h/r$ and $S=r^2$.²¹

The impact of DIC on individual emitters can be seen by the changes in their respective I – V curves before and after cleaning, as shown for PolyNb in Fig. 8. The large shift to the left means a drastic increase of the E_{on} values, both for a

(a)

(b)

FIG. 9. (Color online) FN plots for a typical particulate (a) and scratch (b) on BCP-treated single-crystal Nb sample showing current processing effects.

(a)

(b)

FIG. 8. (Color online) FN plots for a typical particulate (a) and protrusion (b) on electropolished Nb sample before (●, light/orange) and after (●, dark/blue) DIC.

scratch and a particulate. The changes observed in the derived β and S values are as follows: β decreased from 51 to 38 for the particulate and from 148 to 31 for the scratch, while S increased from 1×10^{-15} to $2 \times 10^{-13} \text{ m}^2$ for the former and from 7×10^{-20} to $3 \times 10^{-16} \text{ m}^2$ for the latter. Decreased β and increased S values can be understood by the smoothened edges of the emitters after DIC. It is remarkable that the particulate shows more current fluctuation than the scratch, as expected from their relative mechanical stability.

Often such current fluctuations lead to preliminary deviations from FN behavior, which can usually be minimized by high current processing. The I – V curves for a typical particulate on CryNb [Fig. 9(a)] show: (i) FN behavior with increasing field, which seems to be saturated on reaching currents of few nA, but is finally followed by a current jump; (ii) the curve retraces more stably for decreasing field values. While the stabilized FN fit values ($\beta=48$, $S=9 \times 10^{-19} \text{ m}^2$) suggest an unchanged geometry of the emitter, the preliminary saturation hints for adsorbate effects.²⁵ The I – V curves for a scratch are given in Fig. 9(b), where the emitter has been irreversibly activated at a current of 30 nA and then attains a stable FN behavior ($\beta=61$, $S=3 \times 10^{-19} \text{ m}^2$) in consecutive cycles of increasing and decreasing voltages. In general comparison, the I – V curves of particulates showed more instabilities than surface protrusions.

In total, 54 emitters from all samples have been measured locally for their I – V curves. Representative results for selected emitters are given in Table I, where their characteristic E_{on} , β , and S values before and partially after DIC are

given. The range of E_{on} in MV/m before DIC is 49–84 for Cu and PolyNb, and has been improved by DIC to 63–110 for Cu and PolyNb. Significantly higher values, i.e., a range of 88–167 before and 116–186 after DIC, were found for CryNb and underline the benefit of mirrorlike surfaces for reduced FE. Most of the β values, i.e., 25–56 for Cu, 18–148 for PolyNb, and 17–76 for CryNb, are inversely correlated with the E_{on} values. The derived S parameters cover rather large ranges, i.e., 30 to $3 \times 10^{-6} \mu\text{m}^2$ for Cu and 0.2 to $3 \times 10^{-7} \mu\text{m}^2$ for all Nb samples, most of which are reasonable with respect to the β values. The extreme values, however, cannot be understood within the modified FN theory.

In order to check if grain boundaries cause enhanced FE, the large-grain CryNb samples were scanned (1 mm^2) with $30 \mu\text{m}$ resolution over the grain boundary intersections. No emission was observed up to the highest scanned field levels. Despite the fact that grain boundaries on Nb surfaces can be easily contaminated by segregated impurities, this result suggests that the grain boundary effects play only a minor role for enhanced FE of clean Nb samples.

C. Morphology and composition of emitters

In order to understand the nature and origin of enhanced FE from metallic surfaces, a total of 31 emitters were relocalized and examined with high-resolution SEM and EDX. Figure 10 shows SEM images of various emitters on DIC Cu samples partially with the corresponding EDX spectra. The submicron particulate ($\beta=38$) with sulfur contaminant in Fig. 10(a) seems to be embedded in the surface, and its dark contrast suggests a conducting nature. Similar S contaminations up to $5 \mu\text{m}$ size were found at the emitting sites (β up to 41). Si impurities inside a $10 \mu\text{m}$ large fissure [Fig. 10(b), $\beta=32$] also survived after DIC. Thus, the foreign elements embedded in the surface or trapped inside the grooves could

FIG. 10. (Color online) SEM images with EDX spectra (insets) of FE sites on DIC Cu: (a) sulfur contaminant of submicron size embedded in the surface; (b) silicon impurities inside a fissure; (c) surface irregularity; and (d) large scratch.

not be removed completely by DIC. Moreover, the mark of $10 \mu\text{m}$ size [Fig. 10(c), $\beta=56$] and the large scratch of about $20 \mu\text{m}$ width [Fig. 10(d), $\beta=41$] were identified as emitters with sharp edges and delaminations, thus causing the enhanced FE.

SEM images of some of the emitters found on PolyNb samples are shown in Fig. 11. The particulate of about $50 \mu\text{m}$ length in Fig. 11(a) contains S, Cl, and K and provides a rather small β of 18. Despite its resistance against HPR, it was completely removed by DIC. Usually all the localized field emitting particulates disappeared after DIC treatment of the samples. Surface irregularities like the mark in Fig. 11(b) reflect mishandling of the surface. In one case, a thin flakelike object of about $20 \mu\text{m}$ size [Fig. 11(c)] was

TABLE I. FESM and SEM/EDX results for selected emitters on ten broad area Cu and Nb cathodes. The FN fit parameters are derived for Φ of 4.4 eV (Cu) and 4 eV (Nb). PNB and CNb corresponds to PolyNb and CryNb samples, respectively.

Sample_emitter no.	DIC	E_{on} (MV/m)	FN fit parameters		Element detected by EDX	SEM observation	Related figures
			β	$S(\mu\text{m}^2)$			
Cu1_1		67	25	30	n.m.	n.m.	
Cu1_3		84	41	2×10^{-3}	n.m.	n.m.	
Cu1_2	Y	101	38	7×10^{-4}	S, Cu	$0.6 \mu\text{m}$ embedded particulate	10(a)
Cu1_4	Y	105	32	3×10^{-4}	Si, Cu	Contamination inside fissure	10(b)
Cu2_1	Y	103	41	7×10^{-4}	Cu	Scratch with delamination	10(d)
Cu4_1	Y	110	56	3×10^{-6}	Cu	$10 \mu\text{m}$ mark	10(c)
PNb1_2		54	51	1×10^{-3}	Nb	$20 \mu\text{m}$ particulate	8(a) and 11(c)
PNb1_2 ^a	Y	63	38	2×10^{-1}	Nb	Embedded parts	8(a) and 11(d)
PNb1_3		49	148	7×10^{-8}	Nb	Scratch	8(b)
PNb1_3 ^a	Y	103	31	3×10^{-4}	Nb	Scratch	8(b) and 11(b)
PNb1_4		77	18	5×10^{-1}	S, Cl, K	$50 \mu\text{m}$ particulate	11(a)
CNb1_2		88	71	2×10^{-6}	Nb	No feature found	
CNb3_1		128	48	9×10^{-7}	Nb	$1 \mu\text{m}$ particulate	9(a)
CNb4_1		127	61	3×10^{-7}	Si, Nb	Scratch & $0.5 \mu\text{m}$ particulate	9(b)
CNb4_6		142	59	7×10^{-8}	Nb	$1 \mu\text{m}$ particulate	5
CNb4_5		167	75	4×10^{-9}	Nb	No feature found	5
CNb1_1	Y	116	18.6	3×10^{-2}	Nb	No feature found	
CNb1_5	Y	123	18	6×10^{-2}	Nb	No feature found	
CNb4_2	Y	186	22	4×10^{-3}	Nb	No feature found	

^aSame emitter reidentified after DIC; n.m. means not measured.

FIG. 11. (Color online) SEM images of FE sites on polycrystalline Nb: (a) particulate containing sulfur, chlorine, and potassium (see the inset EDX spectrum) before DIC; (b) large irregularity on the sample surface after DIC; (c) flakelike object before and (d) after DIC, still giving FE.

partially destroyed by DIC and only some embedded part of it survived [Fig. 11(d)] and still emitted, as shown in Fig. 8(b). These facts altogether prove the mechanical and thermal effect of DIC on the particulates for the reduction of FE.

On CryNb samples, the particulates localized as emitters before DIC were always removed by DIC. The SEM images given in Figs. 12(a) and 12(b) prove the removal of a particulate as small as 400 nm in size by DIC from the single-crystal Nb surface. The high-resolution SEM images of the scratch head [Figs. 12(c) and 12(d)] show softened contours of some protrusions, probably due to the mechanical impact

of high-speed dry-ice particles impinging on the surface. It is remarkable that the Si contaminant disappeared after DIC. No FE from this site is observed up to 250 MV/m, i.e., there seems to be no features with β values above about 10. These results show the strength of the DIC technique even for the removal of submicron particulates as well as for the partial smoothing of surface protrusions.

From the observed FE properties and SEM/EDX results (Table I), the emitters can be categorized as follows: (i) particulates with or without contaminants; (ii) features embedded in the surface; (iii) surface protrusions with or without

FIG. 12. (Color online) SEM images of FE sites on single-crystal Nb: scratch with a nearby lying submicron particulate of size ~ 400 nm before (a) and after (b) DIC, high-resolution picture and EDX spectra of the scratch head before (c) and after (d) DIC.

contaminants; and (iv) grooves with trapped contaminants. DIC effectively removes emitters of type (i), and weakens emitters of type (iii), while types (ii) and (iv) are less affected. It should be remarked that 19 out of 51 localized emitters did not show any feature in high-resolution (<5 nm) SEM. This probably hints at the presence of some embedded impurities with low work function.

IV. CONCLUSIONS

DIC has been found to be a very efficient technique for the reduction of FE sites from Cu and Nb surfaces. The remaining emitters found on various FE maps statistically appear at much higher electric fields. The best DIC single-crystal Nb sample did not provide any FE up to 250 MV/m. Selected emitters localized in FESM and identified in SEM have been categorized depending on their shape and impurity content. The I - V curves of most emitters confirm the modified FN theory with reasonable β and S values. In comparison, particulates showed more current instabilities than surface protrusions, as expected from their relative mechanical stability. E_{on} values of all emitters have been significantly improved by DIC. The highest values were achieved for CryNb, i.e., 88–167 MV/m before and 116–186 MV/m after DIC, thus underlining the benefit of mirrorlike surfaces for reduced FE. Large-grain Nb samples do not show any emission from grain boundaries up to 250 MV/m. SEM images of typical emitters prove that DIC removes particulates down to 400 nm size and partially smoothens the edges of protrusions.

ACKNOWLEDGMENTS

We would like to acknowledge A. Aspart and C. Antoine from CEA Saclay for electropolishing of the polycrystalline Nb samples, and A. Brinkmann and J. Ziegler from DESY as well as R. Grimme and C. Zorne from Fraunhofer IPA for experimental support of the DIC apparatus. We are thankful to the Electrical Engineering Department at BUW for access to SEM and EDX facilities. Stimulating discussions with W. Singer and D. Proch from DESY as well as C. S. Pandey from BUW are gratefully acknowledged. The support of the

European Community Research Infrastructure Activity under FP6 “Structuring the European Research Area” program (CARE, Contract Number RII3-CT-2003-506395) is gratefully acknowledged.

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